

# ANALYTICAL ABSTRACTS

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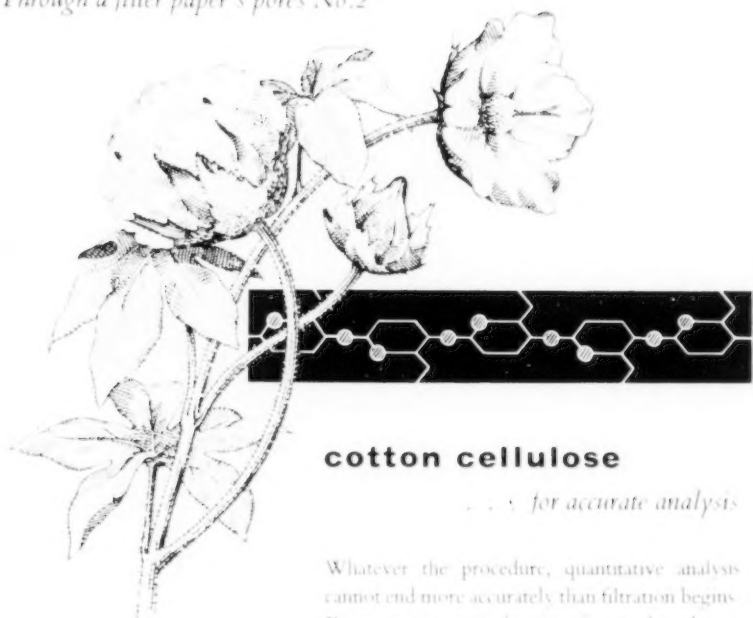
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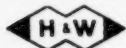
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## ANALYTICAL ABSTRACTS

## 1.—GENERAL ANALYTICAL CHEMISTRY

1082. **Reviews of Russian analytical chemistry.** *Zhur. Anal. Khim.*, 1957, **12** (5), 575-667.—The following review articles are published in a special number. **Growth of analytical chemistry investigations in the USSR**, A. P. Vinogradov, 575-577. **Physico-chemical analysis of analytical systems**, I. V. Tananaev, 578-586. **Use of radioactive isotopes in analytical chemistry**, I. P. Alimarin and N. A. Rudnev, 587-592. **Extraction methods for the separation of elements**, V. M. Vdovenko, 593-599. **Radiometric methods**, V. I. Baranov, 600-605. **Organic reagents**, V. I. Kuznetsov, 606-617. **Spectrographic analysis**, S. M. Raïskii, 618-622. **Electrochemical methods of analytical chemistry**, M. T. Kozlovskii, 623-628. **Photometric methods of analysis**, V. M. Peshkova and I. I. Antipova-Karataeva, 629-636. **Chromatographic analysis**, M. M. Senyavin, 637-646. **Analytical chemistry of the actinides**, P. N. Palei, 647-664. **Inorganic microchemical analysis**, I. M. Korenman, 665-667.

G. S. SMITH

1083. **Methods of analysis by adsorption.** *Anon. Chem. Prod.*, 1957, **20** (9), 379-380.—The use of some selective adsorbents, which operate by the molecular sieve principle, for the analysis and separation of org. compounds is discussed. The synthetic zeolite "Linde Type 5A Molecular Sieve" adsorbs straight-chain aliphatic hydrocarbons, and a method is described for the estimation of these compounds in olefin-free petroleum distillates. The selectivity of silica gel adsorbent for particular stereoisomers, according to the conditions of its prep., is discussed and examples are given of this application.

O. M. WHITTON

1084. **Mechanism of the use of Metanil yellow as an adsorption indicator.** E. Pungor and H. E. Rokosinyi (Inst. f. anorg. u. anal. Chem., L. Eötvös Univ., Budapest). *Z. anal. Chem.*, 1957, **156** (3), 161-169.—In a neutral system the adsorption indicator functions by the formation of the silver salt of the dyestuff, the red colour of which gives the end-point, but in acid solution it acts as an acid - base indicator. The solubility product of the silver salt of the Metanil yellow varies considerably in the two cases and in acid medium the acid - base characteristics of the Metanil yellow change, operating at different pH values with different halides.

F. J. M.

1085. **Indirect potentiometric determination of reducing agents. XXII. Ceric sulphate - ferrous ethylenediamine sulphate method.** Balwant Singh, Sarwan Singh and Harbans Singh (Punjab Univ. Coll., Hoshiarpur, India). *J. Sci. Ind. Res., B, India*, 1957, **16** (4), 173-174.—The excess of ceric sulphate added to the reducing agent, in acid medium, is titrated with ferrous ethylenediamine

sulphate soln. (I), with bright platinum foil as an oxidation - reduction electrode coupled with a S.C.E. through an agar - KCl bridge. Titration values are presented for  $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ ,  $\text{Cu}_2\text{Cl}_2$ , KSCN,  $\text{NaNO}_2$ , Na oxalate,  $\text{H}_2\text{O}_2$ , quinol and pyruvic acid. From the amount of I used, the amount of ceric sulphate required for the oxidation of each substance is calculated.

O. M. WHITTON

1086. **Investigation of the chronopotentiometric method of analysis.** D. J. Fisher (Oak Ridge Nat. Lab., Tenn.). *U.S. Atomic Energy Comm., Rep. CF-55-10-38*, 1956, 18 pp.—In the chronopotentiometric technique, the concn. of a particular ion is determined by measuring the time lag in polarising a quiet mercury-pool cathode when the electrolysing current through the soln. is constant. The limitations of the method are determined by various factors in cell design. The results of studies of these factors are discussed.

NUCL. SCI. ABSTR.

1087. **Purification of lactic acid for chromatographic purposes.** B. K. Preobrazhenskii and O. M. Lilova (V. G. Khlopun Radium Inst., USSR). *Zavod. Lab.*, 1957, **23** (9), 1071.—The impure lactic acid is treated with the calculated amount of zinc oxide suspension, the zinc lactate is filtered off on a Buchner funnel and washed, somewhat less than the calculated amount of conc.  $\text{H}_2\text{SO}_4$  is mixed with the zinc lactate, and the paste containing lactic acid,  $\text{ZnSO}_4$  and water is extracted two or three times with an equal vol. of ether. The required amounts of ZnO and  $\text{H}_2\text{SO}_4$  are obtained from preliminary tests on portions of the sample. The lactic acid obtained still contains traces of Zn and  $\text{H}_2\text{SO}_4$ . It is mixed with a small amount of  $\text{Ba}(\text{OH})_2$  and vacuum-distilled ( $122^\circ$  at 14 mm).

G. S. SMITH

1088. **Recent progress in quantitative paper chromatography.** A. Lacourt (Dept. Microchem., Univ. Brussels, Belgium). *Mikrochim. Acta*, 1957, (3-4), 269-288 (in French).—The use of paper chromatography as a quantitative micro- or ultra-micro technique has been made possible by the direct spectrophotometry of the chromatogram, utilising the transmission of light through the coloured stains. The development of this technique enables the concn. of an element contained in a stain to be rapidly determined. The reproducibility, even with amounts of the order of 0.02 to 0.03  $\mu\text{g}$ , surpasses anything obtained previously. Procedures have been worked out for Al, Mo, Ti, Fe, Ni, Co, Cu, B and U.

L. S. ADLER

1089. **Some features of gas - liquid chromatography.** G. Burrows (Res. Dept., Metropolitan-Vickers Electrical Co. Ltd., Manchester). *Trans. Instn Chem. Engrs*, 1957, **35** (4), 245-257.—This is a description of the method and a discussion of the theory of gas - liquid chromatography.

F. RUMFORD

1090. **Theory of chromatographic methods of gas analysis.** N. M. Turkel'taub and A. A. Zhukhovitskiĭ. *Zavod. Lab.*, 1957, **23** (9), 1023-1034.—A theoretical analysis of the role of certain factors in various types of gas-chromatographic analysis, together with experimental illustrations of the theory, is presented. G. S. SMITH

1091. **Ion-exchange chromatography in quantitative chemical analysis.** M. M. Senyavin (Inst. Geochem. and Anal. Chem., USSR). *Zavod. Lab.*, 1957, **23** (9), 1056-1059.—A review, with 48 references. G. S. SMITH

1092. **Selective ion-exchange sorbents for chromatographic analysis.** E. B. Trostyanskaya and A. S. Tevlina (D. I. Mendeleev Moscow Chem. Tech. Inst.). *Zavod. Lab.*, 1957, **23** (9), 1042-1049.—A review, with 40 references. G. S. SMITH

1093. **Selective ionites and selective ionite membranes.** V. A. Klyachko (Vodgeo Inst.). *Zavod. Lab.*, 1957, **23** (9), 1049-1051.—The use of selective ionite membranes is studied and apparatus for the purpose is described. Recent literature is reviewed. G. S. SMITH

1094. **Separation of complex ions by means of continuous electrochromatography.** C. Bigli and G. Trabonelli (Chem. Inst., Univ. of Ferrara). *Ann. Chim., Roma*, 1957, **47** (6), 743-750.—Mixtures containing  $[\text{Co}(\text{NH}_3)_6]\text{Cl}_2$ ,  $[\text{Co}(\text{NH}_3)_5\text{Cl}]\text{Cl}_2$ ,  $[\text{Co}(\text{NH}_3)_4(\text{NO}_2)_2]\text{NO}_2$  with  $[\text{Co}(\text{NH}_3)_5(\text{NO}_2)]_2$  and  $[\text{Cr}(\text{NH}_3)_6](\text{NO}_3)_3$ ,  $[\text{Cr}(\text{NH}_3)_5\text{Cl}]\text{Cl}_2$ ,  $[\text{Cr}(\text{NH}_3)_4(\text{H}_2\text{O})\text{Cl}_2]\text{Cl}$  with  $[\text{Cr}(\text{NH}_3)_3\text{Cl}_3]$  were separated. The Ni analogues of these compounds are unstable in aq. soln., hence the investigation was confined to the complex  $[\text{Ni}(\text{NH}_3)_6]\text{Cl}_2$  in strongly ammoniacal soln. A. G. COOPER

1095. **Analytical application of -onium compounds. XI. The activities of dodecyltrimethylammonium chloride and dodecylpyridinium chloride as polarographic maximum suppressors.** M. Shinagawa, H. Matsuo, Y. Kameo and Y. Matama (Chem. Dept., Hiroshima Univ., Japan). *J. Sci. Hiroshima Univ.*, A, 1957, **21**, 21-26.—With  $\text{Ni}(\text{NO}_3)_2$  as a negative max. and  $\text{CuSO}_4$  as a positive max. donator the -onium compounds used showed much stronger suppressive activities than gelatin. The reproducibility and precision of measurement of the  $E_{1/2}$  values were satisfactory ( $\pm 10\%$ ), indicating the possibility of using this method for the micro-analysis of max. suppressors. E. S. LANE

1096. **Application of the cathode-ray oscillograph in polarography with alternating current.** J. Heyrovský (Polarographic Inst., Czech. Acad. Sci., Prague). *Chem. Tech., Berlin*, 1957, **9** (5), 257-261.—This is a review of the method and applications of oscillographic polarography. A new apparatus is described which is claimed to be more versatile and accurate than previous instruments. C. A. CURTIS

1097. **Methods of radiochemical analysis.** C. F. Weiss (Inst. of Applied Radioactivity, Leipzig). *Chem. Tech., Berlin*, 1957, **9** (5), 262-266.—Seven different procedures used in radiochemical analysis are described. C. A. CURTIS

1098. **Use of radio-isotopes in quantitative analysis.** A. K. Lavruchina (Inst. Geochem. and Anal. Chem., Acad. Sci., USSR). *Jaderná Energie*, 1957, **3** (9), 272-278.—A review, with 97 references. J. ZÝKA

1099. **Radiometric titrations.** I. P. Alimarin (Moscow State Univ.). *Chem. Anal., Warsaw*, 1957, **2** (4), 297-304.—The method is applicable when the product of the reaction is insoluble or is a complex which may be removed by organic solvent extraction, the ion to be determined, the added ion, or both, being radioactive. At intervals during the titration, samples of the soln. are withdrawn and, after the removal of the ppt. (or complex) by filtration, centrifuging or extraction, the activity is determined, the soln. being subsequently returned to the titration vessel. The end-point, at which a marked change of radioactivity occurs, is obtained as the intersection of two straight lines on a plot of activity vs. vol. (ml) of standard soln. added. A more rapid method, involving the determination of only two points on this curve, is mentioned. Two ions, one of which is radioactive, may be determined simultaneously, provided that the solubilities of the respective ppt. differ appreciably, e.g., titration of  $\text{Ag}^+$  and  $\text{Tl}^+$  (containing radioactive  $^{204}\text{Tl}$ ) with KI. In other cases, a small amount of radioactive ion isomorphous with the ion to be determined may be used as indicator. The radiometric method has been used for the determination of Be (with an accuracy of  $\pm 0.02\%$ ), Zr and Ti in ores and alloys, and a list of other such determinations, together with references, is given. The average titration error is 1 to 2%. T.M.M.

1100. **Use of the electron microscope for microchemical analysis.** L. I. Zemlyanova and Yu. M. Kushnir. *Zavod. Lab.*, 1957, **23** (9), 1083-1087.—A nitrocellulose film is placed on the surface of the soln. to be analysed and a drop of a reagent which will react with the soln. to give crystals is placed on the film. The washed film is examined in an electron microscope. The concn. limits for the detection of certain ions, e.g.,  $\text{Cu}^{2+}$  ( $10^{-8} M$ ) with  $\text{K}_4\text{Fe}(\text{CN})_6$ , are sometimes much lower than those found with optical microscopy. G. S. SMITH

## 2.—INORGANIC ANALYSIS

1101. **Determination of gases in metals by gasometric methods.** R. E. Taylor (Hanford Atomic Prod. Oper., Richland, Wash.). *U.S. Atomic Energy Comm.*, Rep. HW-42663, 1956, 46 pp.—The design and operation of a high-vacuum system for the analysis of gases (N, O and H) present in zirconium, uranium and plutonium are described. Vacuum-fusion and vacuum-extraction results for H normally agree within 5 p.p.m., and values for O are obtained within 10%. NUCL. SCI. ABSTR.

1102. **Use of ion exchange in the analytical chemistry of metals.** S. M. Chernobrov. *Zavod. Lab.*, 1957, **23** (9), 1052-1055.—Papers published since the middle of 1953 are reviewed and 61 references are given. G. S. SMITH

1103. **Adsorption-chromatographic method with complexing agents for the separation of metals.** A. M. Gurvich and T. B. Gapon (Inst. Phys. Chem., USSR). *Zavod. Lab.*, 1957, **23** (9), 1037-1042.—A review, with 16 references. G. S. SMITH

1104. **Analysis for industry [solvent-extraction methods, inorganic chemistry].** T. S. West. *Ind. Chem. Mfr*, 1957, **33**, 420-422.—A survey of recent literature is presented, with particular

reference to the determination of S in steel and the separation of Hf from Zr. (16 references.)

S.C.I. ABSTR.

**1105. Systematic study of solvent extraction with acetylacetone.** A. Krishen (Univ. Pittsburgh, Pa., U.S.A.). *Dissert. Abstr.*, 1957, **17** (5), 970-971. —The extraction behaviour of various metals in acetylacetone and the effect of masking agents were studied. Copper,  $\text{Fe}^{3+}$ , Pb, Th and  $\text{UO}_2^{2+}$  could be determined spectrophotometrically as the acetylacetonate in acetylacetone, and other metals by polarographic or colorimetric methods. Thus,  $\text{Fe}^{3+}$  was separated from  $\text{Th}^{4+}$ ,  $\text{Ce}^{3+}$ ,  $\text{Zn}^{2+}$  and  $\text{Pb}^{2+}$ ;  $\text{Cu}^{2+}$  from  $\text{Ce}^{3+}$ ,  $\text{Zr}^{4+}$ ,  $\text{Zn}^{2+}$  and  $\text{Pb}^{2+}$ ; and  $\text{UO}_2^{2+}$ ,  $\text{Hf}^{4+}$ ,  $\text{Th}^{4+}$  and  $\text{Ce}^{3+}$  from  $\text{Zn}^{2+}$  and  $\text{Pb}^{2+}$ . EDTA inhibited the extraction of  $\text{Cu}^{2+}$ ,  $\text{Zr}^{4+}$ ,  $\text{Hf}^{4+}$ ,  $\text{Th}^{4+}$ ,  $\text{Zn}^{2+}$  and  $\text{Bi}^{3+}$  and so permitted the extraction of  $\text{Fe}^{3+}$ ,  $\text{UO}_2^{2+}$  and  $\text{Ce}^{3+}$ . The possibility of separations in the presence of fluoride, tartrate and 8-hydroxy-7-iodoquinoline-5-sulphonate was also examined. Uranium was quantitatively separated from a 5000-fold excess of Bi. Hafnium was separated from Zr although purity of materials was not high. Aluminium and Cu were separated successfully by multi-stage countercurrent extraction. Successive formation constants for the acetylacetonates of Cu, Pb,  $\text{UO}_2^{2+}$  and Zn were calculated from the extraction data.

O. M. WHITTON

**1106. Chemico-analytical properties of xanthates. IV. Solubility products of zinc, nickel, iron and cadmium xanthates.** A. T. Pilipenko, T. P. Varchenko, E. S. Kudelya and A. P. Kostyushina (Kiev State Univ.). *Zhur. Anal. Khim.*, 1957, **12** (4), 457-461. —The methyl-, ethyl- and butyl-xanthates of Zn, Ni and Cd, the ethylxanthate of Fe and the propylxanthate of Ni are studied, and the solubility products are found. With the compounds of Zn, Ni and Cd, with the exception of nickel methylxanthate, the solubility decreases with increase in the mol. wt. of the radical.

G. S. SMITH

**1107. Investigation of the mechanism of co-precipitation of cations with sulphides by means of radioactive isotopes.** N. A. Rudnev and A. A. Mazur (V. I. Vernadskii Inst. of Geochem. and Anal. Chem., USSR, Moscow). *Zhur. Anal. Khim.*, 1957, **12** (4), 433-442. —The co-pptn. of  $\text{Ga}^{3+}$ ,  $\text{In}^{3+}$  and  $\text{Tl}^{+}$  in acid soln. with  $\text{HgS}$ ,  $\text{CuS}$  and  $\text{CdS}$  is studied. During ageing the co-pptn. may increase ( $\text{Ga}^{3+}$  and  $\text{In}^{3+}$  with  $\text{CuS}$  and  $\text{CdS}$ ), decrease ( $\text{Ga}^{3+}$  and  $\text{Tl}^{+}$  with  $\text{HgS}$ ) or remain unchanged ( $\text{In}^{3+}$  with  $\text{HgS}$  and  $\text{Tl}^{+}$  with  $\text{CuS}$ ).

G. S. SMITH

**1108. Electro-migration on paper in the separation of ions. IV.** Hari Gopal Mukerjee (Scottish Church Coll., Calcutta, India). *Z. anal. Chem.*, 1957, **156** (3), 184-189 (in English). —An electrophoretic method was successfully used to separate Pt and Pd ions from each other using HCl and sodium citrate soln. These elements were also separated from Hg, Cu, Cd, Bi, Fe, Co and Ni.

V. Hari Gopal Mukerjee. *Ibid.*, 1957, **156** (3), 189-193. —The migration sequences of Fe, Al and Be were studied. Separations of Be from Al, and of Fe from Ag, Pb, Hg, Cu, Bi, Zn, Co and Ni in different electrolytes were made. Ferric ions were successfully separated from different binary, ternary and quaternary mixtures and four cations from a mixture of  $\text{Fe}^{3+}$ , Zn, Co and Ni can be readily identified, although under the conditions studied they were not completely separated. F. J. M.

**1109. Radiometric titration of 10<sup>-7</sup> to 10<sup>-5</sup>-gram amounts of transition metals.** J. F. Duncan and F. G. Thomas (Chem. Dept., Univ., Melbourne, Australia). *J. Inorg. Nuclear Chem.*, 1957, **4** (5-6), 376-379. —Two techniques have been used. One involved the use of an isotopic tracer, e.g.,  $^{60}\text{Co}$ ,  $^{203}\text{Hg}$ , and the element was titrated with a soln. of dithione in  $\text{CCl}_4$ . The activity of the aq. phase, at pH  $\approx 1$  for Hg and pH  $\approx 6.7$  for Co, was determined at intervals and the end-point was marked by the disappearance of activity. The results quoted show an error of 0.2  $\mu\text{g}$  on 4  $\mu\text{g}$  of Hg and an error  $> 5\%$  on 2 to 10.1  $\mu\text{g}$  of Co, and  $> 0.09 \mu\text{g}$  on smaller amounts. The second procedure involved the addition of  $^{60}\text{Co}$  to a soln. containing Zn. The titration was performed as before with the aq. phase at pH 5.7 to 6.9. No reduction in the activity was noted until all the Zn had reacted; thereafter the activity decreased, enabling both the Zn and Co to be determined. The error in determining 6.9  $\mu\text{g}$  was  $> 0.05 \mu\text{g}$ , and for amounts  $< 1 \mu\text{g}$  it was  $> 0.065 \mu\text{g}$ .

G. J. HUNTER

**1110. Spectral-isotopic determination of hydrogen in metals.** A. N. Zaidel' and A. A. Petrov. *Vestn. Leningr. Univ.*, 1957, No. 10 (Part 2), 40-51. —Improvements in the method previously described (*Anal. Abstr.*, 1957, **4**, 865) are reported.

G. S. SMITH

**1111. Methods of isotopic analysis of water. V. A float method for density with an accuracy of  $\pm 0.2 \mu\text{g}$  per ml.** A. I. Shtenshtein and E. N. Zvyagintseva (L. Ya. Karpov Phys. Chem. Inst., Moscow). *Zhur. Anal. Khim.*, 1957, **12** (4), 516-522. —To improve the accuracy of the float method, special apparatus for distilling in a current of N, etc., and an accurate thermostat have been designed. Contents of D or  $^{18}\text{O}$  can be determined with an accuracy of  $\pm 0.0002$  atoms %.

G. S. SMITH

**1112. Estimation of moisture and water of crystallisation by azeotropic distillation.** S. Harel and A. Talmi (Fertilizers & Chemicals, Ltd., Haifa, Israel). *Anal. Chem.*, 1957, **29** (11), 1694-1697. —The differentiation between free moisture and water of crystallisation in samples of calcium sulphates and phosphates, and superphosphate fertilisers, has been obtained by azeotropic distillation with benzene and xylene. The method and apparatus are as given by Fetzer (*Anal. Chem.*, 1951, **23**, 1062). Xylene removes both free moisture and water of crystallisation, whereas benzene removes only free moisture. Spurious results can be obtained for samples containing phosphoric acid unless calcium carbonate is added.

G. S. ROBERTS

**1113. Determination of helium in the parts-per-million range.** C. E. Davis, R. H. Hunt and M. J. O'Neal, jun. (Houston Res. Lab., Shell Oil Co., P.O. Box 2527, Tex., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1720. —Helium used as a tracer in gas-flow experiments is determined by a mass spectrometer. The procedure whereby the sensitivity of the instrument is increased by a factor of 1000 from normal is briefly described.

G. S. ROBERTS

**1114. Identification and determination of some alkali metals by paper chromatography.** F. Modreanu, S. Figel and A. Carpov. *Stud. Cercet. Stiint. Chim., Iasi*, 1956, **7** (2), 25-31. —A new



chromatographic method is claimed for the determination of  $\text{NH}_4^+$ ,  $\text{K}^+$ ,  $\text{Rb}^+$ ,  $\text{Cs}^+$  and  $\text{Tl}^+$ . A drop of a soln. of their chlorides, iodides or nitrates (0.05 to 0.2%) is placed on the paper, which is then dried, and the spot is treated with one drop of an acetone soln. of Na picrate (1%). After evaporation of the solvent, the spot is developed with water-saturated nitrobenzene for 3 to 4 hr. The  $\text{Na}^+$  remain immobile, while the other ions form well-defined areas, which are cut out and introduced into small stoppered test-tubes containing 2 ml of acetone. These tubes of the coloured picrates are compared with standard colours. It is claimed that 1 to 5  $\mu\text{g}$  can be thus estimated. H. SHER

**1115. The determination of alkali hydroxides in the presence of alkali carbonates by the precipitation method.** L. Szekeres and E. Bakács-Polgár (Inst. f. allg. Chem. der Landwirtschaftl. Univ., Budapest). *Z. anal. Chem.*, 1957, **156** (3), 194-198.—The Winkler method was found to be inaccurate in 0.1 N soln. and when the  $\text{CO}_3^{2-}$  to  $\text{OH}^-$  ratio is large. Satisfactory results were obtained by substituting  $\text{ZnCl}_2$  for  $\text{HCl}$  and maintaining an excess of  $\text{BaCl}_2$ . Total alkalinity is determined by titrating with acid, with methyl orange as indicator. In a second portion, carbonate is pptd. with excess of  $\text{BaCl}_2$  and the hydroxide remaining is titrated immediately with  $\text{ZnCl}_2$  with phenolphthalein as indicator. Good accuracy is obtained in 0.1 N soln. at  $\text{CO}_3^{2-}$  to  $\text{OH}^-$  ratios of 2:1. F. J. M.

**1116. Mercurimetric determination of alkali-metal tetraphenylborides.** R. Montequi, A. Doadrio and C. Serrano (Inst. "Alonso Barba", Barcelona, Spain). *An. Real Soc. Esp. Fis. Quim.*, B, 1957, **53** (6), 447-452.—The mercurimetric determination of these compounds, especially  $\text{KB}(\text{C}_6\text{H}_5)_4$ , has been studied. A known vol. of 0.1 N mercuric acetate (previously titrated against thiocyanate) is added to the tetraphenylboride, and warmed. After cooling, and acidifying with  $\text{HNO}_3$ , the excess of mercury salt is titrated against aq. 0.1 N  $\text{NH}_4\text{SCN}$ . Since the  $\text{C}_6\text{H}_5\text{Hg}^+$  also react with thiocyanate, 4 equiv. of  $\text{Hg}^+$  correspond to 1 equiv. of alkali metal. Except when the excess of ions to be titrated is >50%, results are accurate, as verified by determinations of K between 1 and 10 mg. C. A. FINCH

**1117. Assay of microgram samples of lithium with a mass spectrometer.** R. E. Sladky (Union Carbide Nuclear Co., Oak Ridge, Tenn.). *U.S. Atomic Energy Comm.*, Rep. Y-1143, 1956, 12 pp.—As little as 0.6  $\mu\text{g}$  of lithium metal as the iodide can be assayed by the procedure presented. An ionisation chamber retort for use with liquid samples is described. Special techniques for preparing and assaying the very small samples are discussed, and the precision of the procedure is given.

NUCL. SCI. ABSTR.

**1118. Spectrophotometric determination of lithium carbide in metallic lithium as the acetylene-silver perchlorate complex.** T. W. Gilbert, jun., A. S. Meyer, jun., and K. C. White (Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1627-1630.—A method is described for the determination of traces of acetylene evolved from lithium carbide, together with hydrogen and ammonia, when metallic lithium is dissolved in water. The method permits the simultaneous determination of the ammonia evolved from the lithium nitride present. The lithium sample,

sealed in an evacuated glass ampoule, is placed in a stainless-steel "bomb" containing water. The bomb is de-aerated with argon and the ampoule broken. When reaction is complete, as indicated by a constant reading on a pressure gauge, the bomb is connected to absorbers containing, respectively, boric acid and silver perchlorate solutions, and the liberated gases are passed through. The u.v. absorption of the acetylene-silver perchlorate complex is measured, and an aliquot from the boric acid soln. is used for the ammonia determination. The method may be used for the range 50 to 2500  $\mu\text{g}$  of acetylene, giving a coefficient of variation of 2.7%. G. S. ROBERTS

**1119. Simple absolute measurement technique for beta radioactivity. Application to naturally radioactive rubidium.** W. F. Libby (Carnegie Inst. of Washington, D.C., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1566-1570.—A simple technique which can be used to measure the half-life of naturally radioactive Rb is described for measuring the absolute radioactivity of solids and liquids by positioning them cylindrically round a Geiger counter. The results agree to within 5% of true absolute assays.

K. A. PROCTOR

**1120. Spectrochemical analysis of caesium in high concentrations of sodium and iron salts.** J. M. Taylor (Hanford Atomic Prod. Oper., Richland, Wash.). *U.S. Atomic Energy Comm.*, Rep. HW-45964, 1956, 11 pp.—A rapid spectrochemical method for determining Cs in high concn. of iron and sodium salts has been developed. Samples are mounted directly on flat-top electrodes, dried and sparked. The standard deviation is  $\pm 2.75\%$ , and the accuracy is within 10% in the range 10 to 200 p.p.m. of Cs. NUCL. SCI. ABSTR.

**1121. Polarographic determination of cuprous ions in copper sulphate solution.** Takuji Ito, Yoshio Hoshino and Keiichi Hara (Tokyo Inst. of Tech., Ookayama). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1957, **60** (6), 692-694.—Cuprous ions in  $\text{CuSO}_4$  in dil.  $\text{H}_2\text{SO}_4$  give an oxidation wave subsequent to the reduction wave of  $\text{Cu}^{2+}$  (vs. the S.C.E.). The wave height changes with  $\text{H}_2\text{SO}_4$  concn. but is proportional to the concn. of  $\text{Cu}^+$  for <0.001 mM. The  $E_p$  value for the oxidation wave of  $\text{Sn}^{2+}$  increases with  $\text{H}_2\text{SO}_4$ , but does not interfere with the determination of  $\text{Cu}^+$  in >5 N  $\text{H}_2\text{SO}_4$ . This method is applicable to the analysis of electrolyte for the anodic dissolution of bronze.

K. SAITO

**1122. Simultaneous complexometric determination of copper and mercury.** K. Ueno (Dojindo & Co., Ltd., Kumamotoshi, Japan). *Anal. Chem.*, 1957, **29** (11), 1668-1669.—Excess of EDTA (disodium salt) is added to the soln. containing  $\text{Hg}^{2+}$  and  $\text{Cu}^{2+}$ , and the unchanged reagent is titrated with standard copper soln. Potassium iodide soln. is then added to decompose the  $\text{Hg}$ -EDTA complex and the liberated EDTA is again titrated with standard copper soln. *Procedure*—To the neutral test soln. containing  $>1 \times 10^{-4}$  g-atom of  $\text{Hg}^{2+}$  and  $\text{Cu}^{2+}$  add EDTA (disodium salt) soln. (15 ml of 0.01 M). Adjust the pH to approx. 10 with aq.  $\text{NH}_3$  (1 M) and add murexide indicator (0.4 g of murexide and 100 g of  $\text{K}_2\text{SO}_4$ ) (0.1 g). Titrate with 0.01 M copper soln. to a colour change from violet to yellow (A ml). Add KI soln. (5%) equiv. to a molar excess of 20 to 30. After a few minutes titrate the soln. again with the standard copper

soln. to the same end-point ( $B$  ml). Then  $Hg$  (mg)  $\equiv B \times 2.006$ , and  $Cu$  (mg)  $\equiv [15 - (A + B)] \times 0.6354$ . Interference is caused by  $Zn$ ,  $Ni$ ,  $Co$ , the alkali metals, nitrate and ferrocyanate.

G. S. ROBERTS

**1123. Separation of copper and nickel by ion-exchange chromatography.** A. P. Kreshkov and E. N. Sayushkina (D. I. Mendeleev Moscow Chem.-Tech. Inst.). *Zhur. Anal. Khim.*, 1957, **12** (4), 559-560.—From a column of a suitable cationite on which  $Cu$  and  $Ni$  have been adsorbed,  $Cu$  can be removed and separated from the  $Ni$  by passage through the column of a soln. containing 5 g of  $NaOH$  and 5 ml of glycerol in 100 ml. Suitable cationites are SDV-2 and -3 (H form); sulphonated coal, KU-1 and SBS are unsuitable.

G. S. SMITH

**1124. Photometric determination of iron in pure and electrolytic copper.** C. M. Dozinell and H. Gill (Hüttenwerke Kayser, Lünen). *Metall.*, 1956, **10** (21-22), 1042-1044.—The sample is dissolved in  $HCl$  and  $H_2O_2$ . A portion equivalent to 1 g of sample is heated with 4 g of cadmium metal and dil.  $HCl$  until the soln. becomes colourless. The soln. is filtered and, when cool, oxidised with two drops of  $H_2O_2$ . Potassium thiocyanate is added and the extinction is measured against water. By comparison with a calibration curve, amounts of  $Fe$  in the range 0 to 100  $\mu g$  can be determined in 1 g of copper. This method is more rapid than that of Gottlieb, in which phenanthroline is used, and is of equal precision. Great care is needed to avoid contamination of the sample and reagents with  $Fe$ . Surface contaminants are removed from the sample and from the cadmium by washing with dil.  $HCl$ .

G. BURGER

**1125. An alkalimetric method for accurate estimation of silver.** A. B. Dutta (Pharm. Training Cent., Jalpaiguri, W. Bengal). *J. Indian Chem. Soc.*, *Ind. Ed.*, 1957, **20** (1), 38-39.—A measured vol. of  $AgNO_3$  soln. is treated with 10%  $Na_2CO_3$  soln. until pptn. is complete. The washed  $Ag_2CO_3$  ppt. is dissolved in 10 ml of a neutralised 10% w/v  $Na_2S_2O_3$  soln., and the liberated alkali is titrated with 0.1  $N$   $HCl$  to methyl orange. Results are claimed to be more accurate than those obtained by Volhard's method. Traces of  $Au$ ,  $Hg$  and  $Mn$  should be absent.

A. M. SPRATT

**1126. Gravimetric separation of alkaline earths.** K. Dreikopf and W. Winzen (Westfälische Berggewerkschaftskasse, Bochum, Germany). *Brennst.-Chemie*, 1957, **38** (13-14), 208-212.—The dissociation pressure of  $SrCO_3$  is much less than that of  $CaCO_3$ , so that mixtures of the two can be analysed by weighing the  $CO_2$  given off under the following conditions. A quartz tube having a side tube for the inlet of dry  $CO_2$ -free  $N$  is swept free from air and the outlet end is connected to a  $CO_2$ -absorption train. A porcelain boat containing  $150 \pm 1$  mg of the mixed carbonates is inserted in the tube and allowed to remain for 1 to 2 min. with a stream of  $N$  passing over it in the reverse direction. It is then pushed into the middle of the tube, which is at  $800^\circ \pm 5^\circ$ , and held at this temp., while  $N$  is passing, for 30 min., which suffices for the complete dissociation of  $CaCO_3$ . The increase of wt. of the absorption train represents the  $CO_2$  originally present as  $CaCO_3$ . If  $Ba$  is present, it should be removed by a double pptn. as  $BaCrO_4$ , and  $Ca$  and  $Sr$  in the filtrate are pptd. as oxalates in the presence of  $NH_4Cl$ . The ppt. is ignited and the  $CaO$  and  $SrO$  are re-dissolved

and re-pptd. as carbonates for the dissociation as described above. Any  $Mg$  is finally pptd. as  $Mg(NH_4)PO_4 \cdot 6H_2O$ . In tests on known mixtures the recoveries of  $Ba$ ,  $Ca$  and  $Sr$  were  $100 \pm 0.5\%$  and of  $Mg \approx 99\%$ .

A. R. PEARSON

**1127. The separation by cation exchange of small quantities of calcium from the products of its neutron irradiation.** M. J. Cabell (A. E. R. E., Harwell, England). A.E.R.E. Report C/M 233, 1957, 8 pp.—Small amounts of  $Ca$  can be separated from microgram amounts of  $^{45}Sc$  and other active impurities by passing the sample soln. ( $> 2$  mg of  $Ca$  in 0.1  $M$   $HCl$ ) through a 4.5-cm column of Zeo-Karb 225 (0.25 g,  $50 \mu$  particle-size) at a constant rate of 0.2 ml per min. The elements are then eluted in the following order: univalent cations, with 1.5  $M$   $HCl$  (1.5 ml);  $Ca^{2+}$ , with 1.5  $M$   $HCl$  (7.5 ml); some bi- and ter-valent cations, with 1.5  $M$   $HCl$  (15 ml);  $Sc$ , with 3  $M$   $HCl$  (10 ml); and some ter- and quadri-valent cations, with 3  $M$   $HCl$  (10 ml).

W. J. BAKER

**1128. Indirect polarographic determination of calcium.** L. Erdey and A. Karsai (Inst. Gen. Chem., Tech. Univ., Budapest). *Acta Chim. Acad. Sci. Hung.*, 1957, **11** (1-2), 171-178 (in German).—Calcium in the concn. range  $6.3 \times 10^{-4}$  to  $2 \times 10^{-3} M$  can be determined by pptn. with a 0.1% aq. soln. of bromanilic acid (**I**) and measuring polarographically the amount of **I** consumed. The reaction and the measurement are carried out in acetate buffer soln. The difference between the height of the peak due to the excess of **I** and that obtained with a blank is proportional to the amount of  $Ca$  in the sample used. The error amounts to  $\pm 2\%$  at concn. of  $10^{-3}$  to  $10^{-2} M$ , but is greater at lower concn. No interference is caused by  $Ag^+$ ,  $Fe^{2+}$ ,  $Al^{3+}$ ,  $Cr^{3+}$  or  $Mg^{2+}$  in concn.  $> 10^{-3} M$ .

G. BURGER

**1129. Ion-exchange methods for the quantitative separation of alkaline earths and their application to the determination of  $^{90}Sr$  in milk ash.** G. M. Milton and W. E. Grummitt (Res. Chem. Branch, Atomic Energy of Canada Ltd., Chalk River, Ontario). *Canad. J. Chem.*, 1957, **35** (6), 541-551.—A study has been made of the effects of column loading and eluting agents on the cation-exchange separation of  $Be$ ,  $Mg$ ,  $Ca$ ,  $Sr$ ,  $Ba$  and  $Ra$ . The principal eluting agents tested were ammonium lactate, ammonium citrate and  $HCl$ . Dowex-50 resin (Ion X, 300 to 400 mesh) was used throughout and the column temp. maintained at approx.  $80^\circ$ . Under optimum conditions, with the use of 0.25 g of resin per milli-equiv. of sample and with ammonium lactate eluent (0.55  $M$  at pH 5 for  $Be$ , and 1.5  $M$  at pH 7 for  $Mg$ ,  $Ca$ ,  $Sr$ ,  $Ba$  and  $Ra$ ), a quant. separation of all six elements in a single sample can be made in 5 hr. This procedure has been applied to the routine determination of  $^{90}Sr$  in bulk-milk ash samples containing 20 mg of  $Sr$  carrier with yields of 85 to 95%. Trace amounts of  $Sr$  occurring naturally in milk have also been determined.

A. JOBLING

**1130. Polarographic determination of strontium.** N. M. Selivanova and G. A. Zubova (D. I. Mendeleev Chem.-Tech. Inst.). *Zhur. Anal. Khim.*, 1957, **12** (4), 466-468.—Concn. of  $SrCl_2 < 0.001 M$  can be determined with the dropping mercury electrode in soln. of 0.1  $N$   $LiCl$  or tetraethylammonium iodide. The only effective maximum suppressor appears to be gelatin, but the amount required increases with the concn. of  $Sr$ . With  $SrSO_4$ ,  $SrCl_2$ ,  $SrSeO_4$  and

Sr acetate the wave heights for the same concn. are the same. With  $\text{Sr}(\text{NO}_3)_2$  the height is twice as great.

G. S. SMITH

**1131. Gravimetric determination of barium in zirconium metal and in certain zirconium salts.** L. Silverman and K. Trego (Atomics Internat. Div., N. Amer. Aviation, Inc., Canoga Park, Calif.). *U.S. Atomic Energy Comm., Rep. NAA-SR-1720*, 1957, 17 pp.—Barium may be determined gravimetrically in hydrated zirconyl chloride and in zirconium metal. Since Zr interferes in the pptn. of Ba as  $\text{BaSO}_4$ , a method is described for the complete separation of the Zr (and Fe) before the final determination of the Ba. The bulk of the Zr is removed by two filtrations at controlled acidities; a cupferron pptn. -  $\text{CHCl}_3$  extraction separates any residual Zr. Gravimetric studies made for the pptn. of Ba in the range 0.2 to 2.0 mg indicated that best results are obtained by the addition of a large excess of  $(\text{NH}_4)_2\text{SO}_4$ . NUCL. SCI. ABSTR.

**1132. Determination of radium in ores and residues.** J. E. Hudgens, R. C. Meyer, C. Zyskowski and L. C. Nelson (New Brunswick Lab., AEC, N.J.). *U.S. Atomic Energy Comm., Rep. NBL-128*, 1957, 20 pp.—A method for the determination of the Ra in ores and uranium-processing residues has been developed. Samples were packed in 12-ml glass vials, the vials sealed and the  $\gamma$ -ray activity of the Ra detected after a growth period of 30 days. A high-pressure ionisation chamber was used as a detector and the  $\gamma$ -rays were filtered through  $\frac{1}{2}$  in. of Pb to minimise the detection of the low-energy part of the Ra  $\gamma$ -ray spectrum. The large sample size used in the method decreases the importance of sample non-homogeneity. Corrections are applied for UX and actinium series  $\gamma$ -rays, and for absorption of Ra  $\gamma$ -rays in the sample material and in the aq. radium standard. The magnitude of the correction for the self-absorption of Ra  $\gamma$ -rays in the sample material was evaluated by using three methods. In the first method, synthetic standards containing known quantities of Ra were used. In the second, the slope of the curve for ion current per gram of sample as a function of the number of grams contained in 12-ml vials was calculated for a large number of samples. In the third, the absorption of Ra  $\gamma$ -rays by representative ores and residues was determined by using a radium source and a sodium iodide crystal scintillation counter. The radium content of a homogeneous sample can be established with a precision equivalent to a standard deviation up to 0.4%. NUCL. SCI. ABSTR.

**1133. Determination of zinc in pigments. Volumetric determination with ferri-ferrocyanide and redox indicator.** J. Faure (La Marine, France). *Peint.-Pigm.-Vern.*, 1957, 33 (4), 340-342.—The determination of  $\text{Zn}^{2+}$  by titration with potassium ferri-ferrocyanide, with a redox indicator, and its application to various zinc pigments (not  $\text{ZnS}$ ) is described. The influence of other ions has been examined;  $\text{Mg}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Ti}^{4+}$  and  $\text{Cr}^{3+}$  do not interfere. Of the interfering ions,  $\text{S}^{2-}$  may be removed by treatment with mineral acid,  $\text{CrO}_4^{2-}$  by reduction with  $\text{SO}_2$ , and  $\text{Cu}^{2+}$  by electrolysis or complexing with  $\text{HCN}$ ;  $\text{Fe}^{2+}$  or  $\text{Fe}^{3+}$  may be removed by pptn. as  $\text{Fe}(\text{OH})_3$ , but there is a risk of entrainment of  $\text{Zn}^{2+}$ , and a better procedure is one based on the rapid and selective dissolution of the Zn (present as  $\text{ZnO}$  or  $\text{ZnCrO}_4$ ) in aq.  $\text{NH}_3$  plus ammonium carbonate. L. A. O'NEILL

**1134. Polarographic determinations of minute quantities of lead in high-purity zinc using the rotated dropping mercury electrode.** Nobuyuki Tanaka and Toshiko Koizumi (Fac. of Sci., Tohoku Univ.). *Bull. Chem. Soc. Japan*, 1957, 30 (3), 303-304 (in English).—The rotating dropping mercury electrode of Stricks and Kolthoff (*J. Amer. Chem. Soc.*, 1956, 78, 2085) was used for the determination of traces of Pb. At 100 r.p.m., at  $25^\circ$ , with a S.C.E. as reference electrode, in 0.1 M KCl containing  $5 \times 10^{-6}$  M polyoxyethylene lauryl ether as max. suppressor,  $i/c$  was constant at 5.8 microamp./ $10^{-4}$  M for concn. of Pb between  $2.94 \times 10^{-7}$  and  $2.94 \times 10^{-4}$  M. Samples of high-purity electrolytic zinc were dissolved in HCl, evaporated to remove excess of acid, and made up to M concn. with max. suppressor. The approx. 0.001% of Pb in the zinc was determined by using the value of  $i/c$  given above and by the standard addition method; the results agreed to within a few per cent.

A. B. DENSHAM

**1135. The amperometric titration of cadmium ions with lithium ferrocyanide solution.** A. Basinski and M. Kuik (Dept. Phys. Chem., Copernicus Univ., Torun, Poland). *Roczn. Chem.*, 1957, 31 (2), 669-675.—Cadmium ions in the presence of  $\text{Li}_2\text{SO}_4$  are titrated directly with standard lithium ferrocyanide solution, by means of the Heyrovský polarograph. The method is applicable to concn. of Cd between 0.0005 and 0.01 M. The error is  $\pm 2\%$ . The titration can be completed in 50 min. The presence of 0.1 M  $\text{H}_2\text{SO}_4$  does not cause interference, but cations that form slightly soluble compounds with  $\text{FeCN}_6^{4-}$ , e.g., Zn, Pb and Cu, and  $\text{K}^+$  and  $\text{NH}_4^+$  interfere. H. DMOWSKA

**1136. A new variation of the sulphide method of cadmium determination.** A. Rokosz (Dept. Inorg. Chem., Jagiellonian Univ., Cracow). *Chem. Anal., Warsaw*, 1957, 2 (4), 345-350.—An investigation into whether the contamination of pptd. CdS is due to autocomplexing or to ion adsorption is described. Pptn. by Berg's hydroxyquinoline method (*Z. Anal. Chem.*, 1927, 71, 321) was used as comparison standard. It is stated that pptn. from soln. containing 0.1 g of  $\text{Cd}^{2+}$  and 0.1 g-equiv. of free  $\text{H}_2\text{SO}_4$  per 100 ml in the presence of surface-active agents showed no improvement in purity. In contrast, results are quoted which show that while ppt. from sulphate soln. containing  $\text{H}_2\text{SO}_4$  are appreciably contaminated, pptn. from perchlorate soln. in the presence of  $\text{HClO}_4$  is comparable in accuracy with the hydroxyquinoline method for concn. of up to 0.2 g of  $\text{Cd}^{2+}$  and 0.06 to 0.1 mole of  $\text{HClO}_4$  per 100 ml of total soln. *Procedure*—Treat 50 ml of the cadmium soln. (in the perchlorate form) with approx. 0.1 mole of  $\text{HClO}_4$  and make up to 100 ml. Heat to boiling and immediately saturate with  $\text{H}_2\text{S}$ . After several hours, filter the soln. through a sintered glass crucible (G4), and wash the ppt. with water, alcohol,  $\text{CS}_2$  and ether. Dry at  $100^\circ$ . T. M. M.

**1137. Volumetric analysis of mercuric salts with EDTA. II. Determination of mercuric sulphide, oxycyanide and salicylate, phenylmercuric acetate and sodium ethylmercurithiosalicylate.** Nobuhiko Iritani and Tomoo Tanaka (Shizuoka College of Pharm., Oshika, Shizuoka). *J. Pharm. Soc. Japan*, 1957, 77 (1), 106-107.—These mercury compounds are readily decomposed with hot HCl or aqua regia and may then be titrated with a mixture of EDTA and  $\text{Mg}^{2+}$  (Iritani *et al.*, *Anal. Abstr.*, 1958, 5, 400). Since organic radicals interfere with the indication



of the end-point of the EDTA titration, organic samples are dissolved in HCl, then treated with  $H_2S$ , and the pptd.  $HgS$  is dissolved in aqua regia before titration.

K. SAITO

**1138. Colorimetric determination of boron - hydrogen compounds with molybdophosphoric acid.** W. H. Hill, J. M. Merrill and R. H. Larsen (Univ., Pittsburgh, Pa.). *U.S. Atomic Energy Comm., Rep. CCC-1024-TR-228*, 1957, 13 pp.—An analytical procedure was developed for the micro-determination of B. The addition of molybdophosphoric acid to decaborane, dimethylamine-borane or sodium borohydride produced blue soln., whose colour intensities were directly related to the content of B. Exact timing of the procedure is necessary because the colour intensity increases with time for decaborane and decreases with time for sodium borohydride.

NUCL. SCI. ABSTR.

**1139. Carminic acid method for spectrophotometric determination of boron.** W. A. Powell and E. H. Poindexter (Univ., Richmond, Va.). *U.S. Atomic Energy Comm., Rep. CCC-1024-TR-226*, 1957, 26 pp.—The use of carminic acid for the spectrophotometric determination of up to  $40 \mu g$  of B has the advantages of sensitivity, reproducibility ( $\pm 2\%$ ), and simplicity. Strong oxidising agents, highly charged cations, and anions that form strong complexes with B are the only ions that interfere seriously. Results of several variable studies led to increased knowledge of and improvements in the carminic acid method. The reagent soln. may be stored either in the light or the dark; the absorbance changes with the age of the reagent soln., but the slope of the calibration curve does not change appreciably. Standardisation of the reagent more often than once a week is not necessary when the blank soln. is used to set the zero of the spectrophotometer.

NUCL. SCI. ABSTR.

**1140. 1:1'-Dianthrime method for spectrophotometric determination of boron.** W. A. Powell and E. H. Poindexter (Univ., Richmond, Va.). *U.S. Atomic Energy Comm., Rep. CCC-1024-TR-229*, 1957, 28 pp.—This method was confirmed as one of the best available for the colorimetric determination of up to  $30 \mu g$  of B. It is three times as sensitive as the carminic acid method, reproducible and simple. Determinations require little time or skill. The effects of variables on the method were studied, and the procedure was improved. Changes in  $H_2SO_4$  concn. do not greatly affect the accuracy of the results. The reagent does not require frequent standardisation if stored in the dark. Highly-charged ions, strong oxidising agents, and ions that form complexes with B are the only ions that interfere.

NUCL. SCI. ABSTR.

**1141. Curcumin (turmeric) method for spectrophotometric determination of boron.** W. A. Powell, E. Hardcastle and E. H. Poindexter (Univ., Richmond, Va.). *U.S. Atomic Energy Comm., Rep. CCC-1024-TR-230*, 1957.—The curcumin method is the most sensitive (four times as sensitive as the 1:1'-dianthrime method) spectrophotometric method available for the determination of B. The method is relatively simple, few steps are involved, there are few sources of error, and the reproducibility is good. The sensitivity is not dependent on the solvent; Beer's law is followed for all solvents. Frequent standardisation is not necessary unless an accuracy greater than 5 or 10% is desired. Oxalic acid increases the sensitivity of the method.

NUCL. SCI. ABSTR.

**1142. Spectrographic determination of boron in graphite.** F. W. J. Garton (U.K. Atomic Energy Authority, Royal Arsenal, London). *Spectrochim. Acta*, 1957, 9 (4), 297-306.—The powdered sample, bound with a phenol-formaldehyde resin, is examined in pellet form, and is arced in graphite holders at 7 amp. d.c. The B I 2497-73-A line chosen is sensitive over the range 0.2 to 1.5 p.p.m. The NO ( $\gamma$ ) line at 2497-17 A is used as an internal standard. Disturbing factors are discussed. Cathode ray display microphotometry, described by Davis and Webb (*cf. Spectrochim. Acta*, 1950, 4, 13) was used to measure concn. of 0.2 p.p.m. and was found to be sensitive down to 0.01 p.p.m. The extension of the method to the determination of B in coke and pitch is discussed.

E. G. CUMMINS

**1143. Determination of tracer quantities of boron in graphite by the method of emission spectrum analysis.** II. S. Held and S. Skalska (Dept. Tech. Phys., Inst. Gen. Chem., Warsaw). *Chem. Anal., Warsaw*, 1957, 2 (4), 327-330.—Standard samples of graphite containing up to  $10 \mu g$  of B per g were prepared and analysed spectroscopically for B by a method described previously by the authors (*Ibid.*, 1956, 1, 294). The standards were then used to construct a working curve of log (intensity) vs. log (B concn.) for the emission of the B analytical line 2497-7 A. About 0.25 g of the sample was placed in each of the copper electrodes and a.c. was used. The intensity of the analytical line changes so sharply with concn. that it allows approximate visual determinations to be made.

T. M. M.

**1144. Determination of boron in titanium alloys by an ion-exchange method.** E. G. Newstead and J. E. Gulbierz (Feltman Ammunition Lab., Picatinny Arsenal, Dover, N.J., U.S.A.). *Anal. Chem.*, 1957, 29 (11), 1673-1674.—A method is described for the complete separation of titanium from boron, by passage through an ion-exchange column. The eluate, which contains the boron, is treated with a soln. of quinalizarin, and the resulting quinalizarin-boron complex is titrated with water to a predetermined colour. The method is capable of measuring a boron concn. with a precision of  $\pm 10\%$  in the 1 to  $10 \mu g$  range. Oxidising ions such as  $NO_3^-$  and  $F^-$  interfere.

G. S. ROBERTS

**1145. Chemical determination of boron in 7% uranium-zirconium and 7% uranium-zircaloy.** J. Rynasiewicz and V. F. Consalvo (Knolls Atomic Power Lab., Schenectady, N.Y.). *U.S. Atomic Energy Comm., Rep. KAPL-M-JR-8*, 1956, 11 pp.—A procedure has been developed for the analysis of B in 7% U-Zr alloys containing approx. 0.015% of B. Fine metal chips from the alloy were fused with  $Na_2CO_3$ . The melt was dissolved in  $H_2O$ , and then made slightly acid. The soln. was made slightly alkaline, filtered, and evaporated to dryness. The B was leached from a slurry of the salt with 95% ethanol, and then determined colorimetrically by the curcumin method.

NUCL. SCI. ABSTR.

**1146. Methods for the analysis of aluminium and aluminium alloys.** 8. Iron (absorptometric 1:10-phenanthroline method). British Standards Institution (2 Park St., London). B.S. 1728: Part 8: 1957, 6 pp.—The alloy is treated with NaOH soln., then acidified with HCl, and undissolved copper is filtered off. After diluting the soln. to volume, an aliquot is taken and the ferrous 1:10-phenanthroline colour is developed by addition of a mixed

reagent containing reducing agent, buffer and 1:10-phenanthroline. The light absorption of the orange-red soln. is measured at about 510 m $\mu$ . The method is suitable for iron contents between 0.05 and 0.6%. The expected degree of reproducibility for pure Al and specified aluminium alloys is indicated by the results presented.

**9. Manganese (volumetric: arsenite-nitrite method).** British Standards Institution. B.S. 1728: Part 9: 1957, 7 pp.—The alloy is dissolved by treatment with NaOH soln. followed by an acid mixture. The Mn is oxidised by (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub> in the presence of AgNO<sub>3</sub> and the HMnO<sub>4</sub> is titrated with arsenite-nitrite soln. The method is suitable for manganese contents between 0.1 and 1.5% and is applicable to alloys containing up to 0.5% of Cr. Results tabulated indicate the expected degree of reproducibility.

**10. Manganese (absorptiometric method).** British Standards Institution. B.S. 1728: Part 10: 1957, 7 pp.—The alloy is dissolved by means of NaOH soln. followed by an acid mixture. The Mn is oxidised to permanganate by boiling with KIO<sub>4</sub>. The light absorption of the soln. is determined at about 525 m $\mu$ . The permanganate is reduced by nitrite and the light absorption of the reduced soln. is deducted. The method is recommended for manganese contents between 0.01 and 0.5%, and is applicable to alloys containing up to 1.0% of Cr. Results presented indicate the expected degree of reproducibility.

O. M. WHITTON

**1147. Separation of gallium from lead and cadmium by the method of ion exchange.** E. P. Tsintsevich and G. E. Nazarova (M. V. Lomonosov Moscow State Univ.). *Zavod. Lab.*, 1957, **23** (9), 1068-1070.—With soln. of pH 8.5 to 9 in the presence of tartrate, and soln. of pH 9 to 10 in the presence of sulphosalicylate, a mixture of Pb and Ga is separated by means of a column of SBS (NH<sub>4</sub><sup>+</sup> form). The Ga passes through and the Pb that is retained can be eluted with ammonium acetate. Separation does not occur with EDTA as complex former. To separate Cd and Ga, tartrate, sulphosalicylate or EDTA can be used under conditions similar to those given above. With mixtures of Pb, Cd and Ga, all three cations are retained on a column of SBS (H form); successive elutions by satd. ammonium acetate soln., 0.2 N HCl and 10% NaOH soln. yield soln. containing only Pb, Cd or Ga. Any Cu and Fe is retained by the column. G. S. SMITH

**1148. Study of certain azo dyes as reagents for indium.** I. M. Korenman, F. R. Sheyanova and R. V. Roshchina (N. I. Lobachevskii Gorki State Univ.). *Zhur. Anal. Khim.*, 1957, **12** (4), 476-480.—Several new colour reactions for In with azo dyes are described, and comparisons with other reagents are made.

G. S. SMITH

**1149. Spectrographic determination of the rare earths.** J. R. Butler (Imperial Coll., London). *Spectrochim. Acta*, 1957, **9** (4), 332-340.—The analysis, by a direct arc method, of rare-earth oxides separated from minerals is described. A dispersion of 0.27 m $\mu$  per mm at 400 m $\mu$  was obtained with a glass Littrow prism in a specially constructed spectrograph. This is sufficient for the determination of all the 16 naturally occurring rare earths in oxides poor in Ce, and of all except Eu, Ho and Tm in oxides rich in Ce. Use of a grating instrument is discussed. E. G. CUMMINS

**1150. Determination of rare earths in thorium.** E. J. Center, W. M. Henry and R. D. Householder (Battelle Memorial Inst., Columbus, Ohio). *U.S. Atomic Energy Comm., Rep. BMI-260*, 1957, 13 pp.—A quant. method for the determination of individual rare earths in thorium down to a level of 0.05 p.p.m. is described. The procedure comprises a chromatographic cellulose-column separation followed by a solution-type spectrographic determination. Values are given for the recovery of several rare earths.

NUCL. SCI. ABSTR.

**1151. Preparation of radiochemically pure cerium by solvent extraction.** H. W. Kirby (Monsanto Chemical Co., Miamisburg, Ohio, U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1599-1601.—The preparation from a saturated aq. soln. of reagent-grade cerous nitrate is carried out by extraction with tri-*n*-butyl phosphate. Thorium is also extracted, but cerium can be separated from it by an extraction of the organic phase with 8 N HNO<sub>3</sub>. The product contained 80% of the cerium and < 2% of the original radioactivity.

K. A. PROCTOR

**1152. Analytical studies of the fluorescence of samarium in calcium sulphate.** C. G. Peattie and L. B. Rogers (M.I.T., Cambridge, Mass., U.S.A.). *Spectrochim. Acta*, 1957, **9** (4), 307-322.—Microgram amounts of Sm are detectable fluorimetrically when, after co-pptn. with CaSO<sub>4</sub>, the Sm<sup>III</sup> is reduced to Sm<sup>II</sup> by being exposed to a beam of 3-MeV electrons from a Van der Graaff accelerator. Interference of trivalent rare earths is discussed. Under similar conditions for the pptn. and electron bombardment, about  $6 \times 10^{-2}$   $\mu$ g of Eu can be detected by using 253.6-m $\mu$  radiation instead of the 365-m $\mu$  radiation used for Sm. E. G. CUMMINS

**1153. Determination of microgram quantities of carbon by low-pressure combustion.** J. H. Edgerton and H. G. Davis (Oak Ridge National Lab., Tenn.). *U.S. Atomic Energy Comm., Rep. ORNL-2211*, 1957, 12 pp.—The determination of microgram quantities of C in alloys by the low-pressure combustion method is based on the combustion of the specimen in O at 135 mm pressure, freezing out the CO<sub>2</sub>, pumping out the excess of O, and then measuring the CO<sub>2</sub> in the gas phase with a known volume by means of a McLeod gauge. The recommended concn. range is from 0.003 to 0.3 mg of C in a 0.3-g sample, i.e., from 0.001 to 0.1% of C. The relative standard deviation of the method is 10%. The unique feature of the apparatus is the use of a Toepler pump to transfer the CO<sub>2</sub> to the McLeod gauge.

NUCL. SCI. ABSTR.

**1154. Determination of silicon in the presence of phosphorus, iron, copper, manganese and titanium.** V. N. Galakhova (Stalin Medicinal Inst., Donbass). *Zhur. Anal. Khim.*, 1957, **12** (4), 499-503.—The use of the vanadomolybdic complex is unsatisfactory for the determination of Si in the presence of P, Fe, Cu, Mn and Ti.

G. S. SMITH

**1155. Photometric determination of small silicon contents in cryolite and other water-insoluble fluorides.** W. Augustyn and Z. Sosin (Inst. Inorg. Chem., Gliwice, Poland). *Chem. Anal., Warsaw*, 1957, **2** (4), 305-314.—Methods are described for preparing soln. of the fluorides for analysis, and experimental details are given for the photometric determination of Si in the yellow molybdosilicate soln. by using a Pulfrich photometer and a standard curve obtained with K<sub>2</sub>CrO<sub>4</sub> soln. according to the

method of Dobkina (*Zavod. Lab.*, 1948, **14**, 755). The determinations were carried out on samples containing known amounts of  $\text{SiO}_2$ . The method was then applied to commercial samples of cryolite and calcium fluoride: the relative error for these is given as 2 to 12%. The time for a single determination is 30 min. with dissolution in  $\text{HNO}_3$ ; 1.5 to 2 hr. with alkaline fusion. T. M. M.

**1156. Spectrographic scheme for the determination of aluminium, titanium, iron, calcium, magnesium and manganese in silicates.** B. J. Rushton and G. D. Nicholls (Dept. of Geol., The Univ., Manchester, England). *Spectrochim. Acta*, 1957, **9** (4), 287-296. —Low-voltage d.c. anodic excitation in a carbon arc is employed. The more volatile group (Fe, Mg, Ca and Mn) is standardised by Sr. with arcing conditions slightly different from those for the less volatile pair, Al and Ti, which are standardised against Y. E. G. CUMMINS

**1157. Determination of tin in zircaloy and uranium - zircaloy: colorimetric procedure.** J. L. Marley and O. J. Articulo (Knolls Atomic Power Lab., Schenectady, N.Y.). *U.S. Atomic Energy Comm., Rep. KAPL-M-JLM-2*, 1957, 6 pp. —Tin is separated from Zr on a Dowex-2-X ion-exchange column in HCl. The Sn is eluted with dil.  $\text{H}_2\text{SO}_4$  and determined spectrophotometrically with dithiol. Diethylammonium dithiocarbamate is used to extract Sn from Zr-U mixtures before the dithiol determination. NUCL. SCI. ABSTR.

**1158. High-frequency titration of lead.** D. T. Sawyer and P. S. Farrington (Univ. of California, Los Angeles, U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1688-1689. —The titration of  $\text{Pb}^{2+}$  with  $\text{K}_2\text{Cr}_2\text{O}_7$  soln., with a high-frequency oscillator to detect the end-point, is described. An accuracy within 0.2% is to be expected. G. S. ROBERTS

**1159. Determination of the isotopic composition of lead.** S. I. Z̋ykov and N. I. Stupnikova (V. I. Vernadskii Inst. of Geochem. and Anal. Chem., USSR). *Zhur. Anal. Khim.*, 1957, **12** (4), 556-558. —The Pb is obtained in the form of  $\text{PbI}_2$ , and with  $\text{Cu}_2\text{I}_2$  as carrier is analysed by means of a mass spectrograph. G. S. SMITH

**1160. Polarographic determination of lead, copper and zinc in pyrites, marcasite and spent oxides.** K. Lasiewicz and H. Zawadzka (Anal. Dept., Inst. of Sulphuric Acid and Phosphorus Fertilisers, Luboń, Poznań). *Chem. Anal., Warsaw*, 1957, **2** (1), 22-28. —Procedure for Cu and Zn—Weigh two 0.2-g samples. To one add standard soln. of Zn (10 mg) and Cu (10 mg), then treat each sample with Lefort's mixture ( $\text{HNO}_3$ -HCl, 3:1, v/v) (12 ml) and  $\text{KClO}_3$  (1 g) and evaporate to dryness; add HCl (1:1) (50 ml) and again evaporate. Dissolve the residue in HCl (1:1) (3-2 ml) and water (50 ml), add 0.2% gelatin soln. (5 ml) and pyridine (2-4 ml), and dilute to 100 ml. Filter, mix the filtrate (20 ml) and pyridine (3 ml), de-oxygenate and polarograph between 0 and 1.3 V. Procedure for Pb—Weigh two 0.5-g samples; to one add a standard soln. of Pb (20 mg), and to both, Lefort's mixture (12 ml) and  $\text{KClO}_3$  (1 g), and evaporate to dryness. To the residue add HCl (1:1) (50 ml) and evaporate, then add HCl (1:1) (2 ml) and water (50 ml). Boil the soln. and treat it with 10% hydroxylamine hydrochloride soln. (10 ml). When colourless add HCl (1:1) (18 ml) and 0.2% gelatin soln. (5 ml), and

dilute to 100 ml. After de-oxygenation, polarograph between 0 and 1 V. The results obtained with concn. of the three metals from 0.3 to 10% agreed well with those obtained by the chromate method for Pb, the iodimetric method for Cu, and the phosphate method for Zn; better precision was attained by the polarographic method. Cobalt interfered with the determination of Zn and a correction had to be applied. K. F. SPOREK

**1161. Estimation of titanium in beach sands.** J. A. Corbett and D. H. Parkhurst (Univ. Melbourne, Australia). *Proc. Aust. Inst. Min. Metall.*, 1957, (182), 55-65. —A differential colorimetric technique, using  $\text{H}_2\text{O}_2$  as reagent with a solution of the sample in  $\text{H}_2\text{SO}_4$ , is described for the estimation of Ti in beach sands and their ore-dressing products. An accuracy of  $\pm 0.3\%$  is obtained for concentrates containing >95% of  $\text{TiO}_2$ , and  $\pm 1\%$  for tailings containing 0.2% of  $\text{TiO}_2$ . S.C.I. ABSTR.

**1162. Analysis of titanium dioxides.** W. Lamprecht (Inst. für Lackforschung, Giessen, Germany). *Farbe u. Lack*, 1957, **63**, 342-345. —The  $\text{TiO}_2$  is treated with KI in methanol and exposed to daylight for 10 hr., after which the mixture is diluted with  $\text{H}_2\text{O}$ , the pigment filtered off, and the iodine determined colorimetrically with addition of starch. The iodine liberated is proportional to the anatase to rutile ratio in simple mixtures of the two forms of  $\text{TiO}_2$ . Other white pigments must normally be separated from the  $\text{TiO}_2$  before application of the method. L. A. O'NEILL

**1163. Detection of a micro amount of zirconium with an ion exchanger.** Kiyoshi Kato, Takeo Murase and Hidetake Kakihana (Chem. Dept., Fac. of Sci., Nagoya Univ., Chikusa-ku). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1957, **78** (6), 854-859. —The detection of Zr was studied with the ion-exchange resins Amberlite IRC-50 and IR-120, and Dowex 50-X1, X8(W), 1-X1 and 1-X8 impregnated with 1-nitroso-2-naphthol, 2-nitroso-1-naphthol, morin, alizarin or alizarin red S (I). The use of Dowex 1-X1 ( $\text{SO}_4$ ) containing I gives the most satisfactory results. Dowex 1-X1 is treated with 0.1% I in methanol for 30 min. and put into one drop of the sample soln. of pH 1.6 to 1.8 containing 0.1 to 1 M  $\text{SO}_4^{2-}$ . The dark-brown grains turn purple in the presence of > 0.03  $\mu\text{g}$  of Zr (orange in its absence) within 24 hr. (in the presence of > 0.8  $\mu\text{g}$ , within 30 min.). Interference results from the presence of > 10 times the weight of  $\text{VO}_2^+$ ,  $\text{MoO}_4^{2-}$ ,  $\text{WO}_4^{2-}$ ,  $\text{F}^-$ ,  $\text{UO}_2^{2+}$  and oxalate, and of > 100 times the weight of  $\text{Fe}^{3+}$ , Sb, Bi, Ti and  $\text{SiO}_3^{2-}$ . K. SAITO

**1164. Ion-exchange spectrophotometric determination of thorium.** O. A. Nietzel, B. W. Wessling and M. A. DeSesa (Nat. Lead Co., Inc., Raw. Mat. Dev. Lab., Winchester, Mass.). *U.S. Atomic Energy Comm., Rep. WIN-62*, 1957, 16 pp. —A sensitive method of analysis was needed for the determination of Th in connection with studies on the production of Th-free uranium concentrates. Two ion-exchange procedures were developed for the separation of Th before spectrophotometric determination with thorin reagent. In the anion-exchange procedure, possible cation interferences are removed by adsorption of their chloro complexes from 9 M HCl on to Dowex-1 resin. The more specific cation-exchange method consists in adsorption of cations from the sample on to Amberlite

IR-120 resin, elution of most of the cations with 2 M HCl, and recovery of the Th by elution with 3 M  $\text{H}_2\text{SO}_4$ . The developed procedures were successfully used to determine Th in a variety of samples and should find application in the determination of low concn. of Th in other complex materials.

NUCL. SCI. ABSTR.

**1165. Versene titration of thorium and aluminium.** W. R. Cornman (Du Pont de Nemours & Co., Savannah River Lab., Augusta, Ga.). *U.S. Atomic Energy Comm.*, Rep. DP-171, 1956, 8 pp.—A method was developed for the determination of Th and Al in  $\text{HNO}_3$  soln. containing small amounts of  $\text{F}^-$ . Titration with Versene (EDTA, disodium salt) was employed, with alizarin red S as an internal indicator.

NUCL. SCI. ABSTR.

**1166. New gravimetric methods for the determination of thorium, aluminium, beryllium and zinc, and their separation from other elements.** G. Spacu and T. I. Pirtea (Univ., Bucharest, Romania). *Rev. Chim., Romania*, 1956, **1** (2), 5-25 (in French).—A 10% (w/v) soln. (pH 8) of Na mercaptobenzothiazole (**I**) can be used for the rapid macro- or micro-gravimetric determination of Th, Al, Zn and Be in neutral or slightly acid soln. The cryst. ppt. formed almost immediately on the addition of an excess of **I** to the sample soln. (5 to 50 ml, 0.003 to 0.1 g) is filtered off quickly, washed with a dil. soln. of **I**, dried at  $105^\circ$  to  $120^\circ$ , and weighed as  $\text{Th}(\text{C}_7\text{H}_4\text{NS}_2)_4$ ,  $\text{Al}(\text{C}_7\text{H}_4\text{NS}_2)_3$ ,  $\text{Zn}(\text{C}_7\text{H}_4\text{NS}_2)_2$  or  $\text{BeO}$ . The reagent is prepared by adding slightly less than the stoichiometric amount of  $\text{N NaOH}$  to mercaptobenzothiazole, and removing the excess of the thiazole by filtration. The necessary modifications for the determination of each element, and its separation from Mg, Al and Fe, are given. In the determination of Al the reaction is sensitive to as little as 4  $\mu\text{g}$  of Al; Mg (e.g., in soln. of Mg-Al alloy) does not interfere and can be determined as 8-hydroxyquinolate in the filtrate in the presence of, or after removing, the excess of **I**. Alkali, alkaline-earth elements and Mg do not affect the determination of Zn, but if acetate or tartrate (added to mask Al or Fe or both) is present the ppt. should be ignited to  $\text{ZnO}$ . Beryllium can be determined alone or in the presence of  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{NH}_4^+$ ,  $\text{Mg}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{SO}_4^{2-}$ ,  $\text{NO}_3^-$ ,  $\text{Cl}^-$ ,  $\text{Br}^-$ ,  $\text{I}^-$  or  $\text{CH}_3\text{CO}_2^-$ ; Mg can be determined as 8-hydroxyquinolate in the filtrate after pptn. of Be, but the Al should be pptd. with 8-hydroxyquinoline and the Be then determined in the filtrate after concn. and neutralisation with aq.  $\text{NH}_3$ . The accuracy of the method is high because pptn. of the metal salt of mercaptobenzothiazole is quant. between  $15^\circ$  and  $25^\circ$ .

W. J. BAKER

**1167. Separation of thorium and uranium nitrates by anion exchange.** D. J. Carswell (A.E.R.E., Harwell, England). *J. Inorg. Nuclear Chem.*, 1957, **3** (6), 384-387.—The method depends on the formation of anionic nitrate complexes by Th;  $^{230}\text{U}$  and  $^{232}\text{Th}$  are eluted successively at  $77^\circ$  by 4 M  $\text{HNO}_3$  from a column of De-Acidite FF resin. Preparation of the resin involves cycling it through the hydroxyl and nitrate forms and final conditioning with 6 M  $\text{HNO}_3$ .

G. J. HUNTER

**1168. Determination of the phosphorus pentoxide content of bauxite.** L. Erdey and V. Fleps (Inst. Gen. Chem., Tech. Univ., Budapest). *Acta Chim. Acad. Sci. Hung.*, 1957, **11** (1-2), 195-203 (in German).—The phosphate is extracted with isobutyl alcohol as molybdophosphate, which is reduced by

shaking the extract with  $\text{SnCl}_2$  soln., and the extinction of the resulting blue soln. is measured. In the amount present in bauxite, Fe, Al,  $\text{SiO}_2$ , Ti and V do not interfere. In the range 0.1 to 0.7%,  $\text{P}_2\text{O}_5$  can be determined with an accuracy of  $\pm 5\%$ .

G. BURGER

**1169. Determination of microgram quantities of tetrasodium pyrophosphate by the paper-impregnation method.** W. Kielczewski (A. Mickiewicz Univ., Poznań). *Chem. Anal., Warsaw*, 1957, **2** (4), 336-339.—The area of the white spots formed by the pyrophosphate soln. on paper impregnated with copper ferrocyanide is measured and compared with a standard (straight-line) curve obtained by using the same volumes of soln. of known pyrophosphate content on identically prepared paper. The presence of orthophosphate does not cause interference and the error does not exceed 2%. Full details of the preparation of the paper and of the determination are given.

T. M. M.

**1170. Rapid determination of arsenic in ores and other non-metallic materials and in nitric acid-resisting ferro-alloys.** H. Ploum (Max-Planck-Inst. Eisenforsch., Düsseldorf, Germany). *Arch. Eisenhüttenw.*, 1956, **27**, 761-766.—When samples containing  $\text{As}^{III}$  are dissolved in  $\text{HNO}_3$  and  $\text{HF}$ , losses occur by evaporation. Instead of this method, an oxidising melting process is applied in which the As is separated by distillation from a  $\text{ZnCl}_2$  soln. and absorption in hydrous ferric oxide. The As is then determined potentiometrically. Samples of about 1 g are sufficient.

CHEM. ABSTR.

**1171. Rapid method for the determination of the total arsenic in commercial calcium arsenate by means of an ion exchanger.** R. Kling and J. Lindeman (Inst. Inorg. Chem., Gliwice, Poland). *Chem. Anal., Warsaw*, 1957, **2** (4), 331-335.—The method was suggested by one due to Odencrantz and Rieman (*Anal. Chem.*, 1950, **22**, 1066). It was compared with the classical reduction-distillation method and found satisfactory, although when using standard soln. of arsenic the relative error was approx. -3%. The sample of commercial calcium arsenate was evaporated with  $\text{HNO}_3$  and  $\text{KBrO}_3$ , the residue dissolved in conc. HCl, diluted and passed through a cationite column. The arsenate in the eluate was determined iodimetrically. The time required was half that for the classical method. Omission of the ion-exchange stage led to inaccurate results.

T. M. M.

**1172. Vanadometry. Estimation of arsenite. Use of iodine monochloride and osmium tetroxide as catalysts.** K. Bhaskara Rao, H. S. Gowda and G. Gopala Rao (Andhra Univ., Waltair, India). *Z. anal. Chem.*, 1957, **156** (3), 180-184 (in English).—*Osmium tetroxide*—An aliquot of arsenite soln. is added to a known excess of standard vanadate soln., heated on the water bath for 2 to 5 min. with 1 ml of dil.  $\text{H}_2\text{SO}_4$  (1:3), then cooled;  $\text{H}_2\text{SO}_4$  is added to  $\text{N}$  concn. and the soln. is titrated with ferrous ammonium sulphate, with diphenylbenzidine as indicator. *Iodine chloride*—Sodium vanadate soln. (0.05 N) (15 ml), 10 ml of 0.01 M arsenite, and 1 ml of 0.005 M ICl in 50 ml of soln. 8 N in  $\text{H}_2\text{SO}_4$  are allowed to react for 20 min. The excess of vanadate is titrated with ferrous ammonium sulphate, with N-phenylanthranilic acid as indicator. Both methods give a very high degree of accuracy. (Cf. *Anal. Abstr.*, 1958, **5**, 455.)

F. J. M.



**1173. Volumetric determination of bismuth in the presence of phosphates, sulphates, oxalates, salicylates and heavy metals.** M. B. Shchigol' (Kiev Medical Inst.). *Zhur. Anal. Khim.*, 1957, **12** (4), 481-484.—The soln. containing Bi, other heavy metals, sulphates, etc., is boiled and treated with excess of 0.1 N  $\text{Na}_2\text{HPO}_4$ . After 2 min. the ppt. of  $\text{BiPO}_4$  is filtered off and washed with hot water. The ppt. is dissolved in 10 to 15 ml of 0.1 N  $\text{H}_2\text{SO}_4$  and 0.5 to 1 g of KI. After dissolution of the ppt. and boiling for a further 2 min., 0.4 N Na oxalate is added from a burette to decolorise the soln., 5 g of KCl is added, the soln. is cooled and neutralised to methyl red with 0.1 N NaOH, the oxalate complex is decomposed by boiling with a measured amount of 0.1 N NaOH, and the excess of alkali is titrated in the cooled soln. with 0.1 N  $\text{H}_2\text{SO}_4$ , phenolphthalein being used as indicator. In the presence of Pb the method must be modified. The soln. in dil.  $\text{HNO}_3$  is treated with excess of saturated  $\text{K}_2\text{SO}_4$  soln., the ppt. of  $\text{PbSO}_4$  and  $\text{Bi}_2(\text{SO}_4)_3$  is treated with KI, which reacts with  $\text{Bi}_2(\text{SO}_4)_3$  but not with  $\text{PbSO}_4$ , and the filtrate is treated with Na oxalate, etc. G. S. SMITH

**1174. Permanganometric determination of vanadium in ferrovanadium after reduction with sodium nitrite.** L. Erdey and K. Vigh (Inst. Gen. Chem., Tech. Univ., Budapest). *Acta Chim. Acad. Sci. Hung.*, 1957, **11** (1-2), 73-83 (in German).—Vanadium in ferrovanadium can be reduced to  $\text{V}^{IV}$  with  $\text{NaNO}_2$  and titrated with  $\text{KMnO}_4$ . The sample (0.25 g) is dissolved in a mixture of  $\text{H}_2\text{SO}_4$  (1:1) (50 ml) and  $\text{HNO}_3$  (1:3) (20 ml) and boiled until fumes of  $\text{SO}_3$  appear. After cooling the soln. and diluting to 200 ml,  $\text{NaNO}_2$  (1 g) is added and the soln. is shaken and set aside for 10 min. Urea (1.5 g) is added, the soln. is shaken until effervescence ceases, then heated to between 60° and 70°, and the vanadium is titrated with 0.1 N  $\text{KMnO}_4$ . The procedure can be carried out in 1 hr. The standard deviation, from 12 determinations, was  $\pm 0.03$  ml of 0.1 N  $\text{KMnO}_4$ . G. BURGER

**1175. Determination of certain trace impurities in graphite by radioactivation. I. Vanadium.** A. A. Smales and D. Mapper (A.E.R.E., Harwell, England). A.E.R.E. Report C/R 2392, 1957, 15 pp.—The sample (up to 200 mg) is irradiated in a pile for 4 min. together with a standard of  $\text{V}_2\text{O}_5$  in sucrose as diluent. The decay of the 3.7-min.  $^{52}\text{V}$  is then followed by using standard  $\beta$ -counting equipment. The content of V of the sample is calculated from the relative activities of the sample and standard. On two series of six determinations on samples containing 141 and 12.0 p.p.m. of V, standard deviations of  $\pm 7$  and  $\pm 0.35$  p.p.m., respectively, were recorded. G. J. HUNTER

**1176. Use of ascorbic acid in amperometric titration. I. Determination of vanadium and cerium in the presence of other elements.** Z. A. Gallaï, V. G. Tiptsova and V. M. Peshkova (M. V. Lomonosov Moscow State Univ.). *Zhur. Anal. Khim.*, 1957, **12** (4), 469-475.—The  $\text{E}_1$  for the oxidation of ascorbic acid ( $4.5 \times 10^{-4}$  N) on a rotating platinum electrode is  $+0.38$  V at pH 6.81 (phosphate buffer),  $+0.68$  at pH 4.60 (acetate buffer),  $+0.92$  in 0.1 N  $\text{H}_2\text{SO}_4$  and  $+1.00$  in 1 N  $\text{H}_2\text{SO}_4$ . In 0.1 N  $\text{H}_2\text{SO}_4$  the diffusion current is proportional to concn. up to  $10^{-3}$  M. For amperometric titration with ascorbic acid, a soln. (0.05 N) is prepared by dissolving 4.4 g in water, adding 4 ml of 75% formic acid and 0.1 g of EDTA (disodium salt) and diluting to

1 litre. It is standardised potentiometrically against  $\text{KIO}_3$ . To determine  $\text{V}^{5+}$ , titration is carried out in 0.1 N to N  $\text{H}_2\text{SO}_4$  at  $+0.9$  V. With small amounts of V (0.04 mg) in 10 ml of 0.1 N  $\text{H}_2\text{SO}_4$ , a 0.005 N soln. of the reagent can be used. No interference is caused by Ni, Mn, Zn, Al and Cr, or by Mo at a Mo to V ratio of  $>20:1$ . The content of W must not exceed that of V. To separate large amounts of Fe, the acid soln. containing  $\text{H}_2\text{O}_2$  is passed through a column of SBS (H form), which is then washed with a 1% soln. of  $\text{H}_2\text{O}_2$  in 0.1 N  $\text{H}_2\text{SO}_4$ . The excess of  $\text{H}_2\text{O}_2$  in the filtrate is decomposed by  $\text{KMnO}_4$  and the excess of  $\text{KMnO}_4$  is destroyed by oxalic acid. To determine V in steel, the soln. in acid is treated with aq.  $\text{NH}_3$  until a ppt. forms, a few drops of N  $\text{H}_2\text{SO}_4$  are added to dissolve the ppt., the Fe is separated as described above, and the amperometric titration is carried out. The titration of Ce is carried out at potentials between  $-0.1$  and  $+0.5$  V in 2 N to 10 N  $\text{H}_2\text{SO}_4$ . In 5 N  $\text{H}_2\text{SO}_4$ , Ce can be determined at  $+0.3$  to  $+0.5$  V without interference from Fe. After the titration, Fe can be determined by making the soln. nearly neutral, heating to between 40° and 60°, and titrating at zero potential. G. S. SMITH

**1177. Annotated bibliography of the analytical chemistry of niobium and tantalum, Jan. 1935 to June 1953.** F. Cuttitta. *U.S. Geol. Survey Bull.*, 1957, (1029 A), 1-73.

**1178. Determination of protactinium-233.** F. L. Moore and S. A. Reynolds (Oak Ridge National Lab., Tenn., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1596-1599.—2,6-Dimethylheptan-4-ol has been found to be a suitable solvent for extracting  $^{233}\text{Pa}$  from  $^{233}\text{U}$ -recovery process soln. The sample soln. should be adjusted to 6 M HCl and contain 4% of oxalic acid to inhibit the extraction of  $^{90}\text{Nb}$ . If the sample contains Th, a preliminary extraction is made without oxalic acid. K. A. PROCTOR

**1179. Colorimetric apparatus for investigating gases. I. Continuous determination of small oxygen contents.** S. Waszak (Inst. of Gen. Chem., Warsaw). *Chem. Anal., Warsaw*, 1957, **2** (4), 376-384.—The principle of the method is due to Brady (*Anal. Chem.*, 1948, **20**, 1033) and depends on the reversible reaction  $9:10$ -anthradiol-2-sulphonic acid (red)  $+ \text{O} \rightarrow$  anthraquinone-2-sulphonic acid (colourless) which is rapid and quant. The intensity of the colour is determined photometrically and registered automatically, giving a measure of the oxygen absorbed. Acidic gases ( $\text{CO}_2$ ,  $\text{H}_2\text{S}$ , etc.),  $\text{CS}_2$ , and CO above a certain limit, interfere. Full details, with a schematic diagram and a photograph of the apparatus are given. T. M. M.

**1180. Micro-determination of sulphur according to Zimmermann.** A. Dirscherl (Microanal. Lab., E. Merck A.-G., Darmstadt). *Mikrochim. Acta*, 1957, (3-4), 421-426 (in German).—High values may be obtained in the titrimetric Zimmermann method when the modified procedure without the use of a retarding capillary is adopted. This is because even relatively brief exposure of the reaction mixture to unfavourable pH ranges during the oxidation of CdS to elementary S can result in this oxidation going too far. If the mixture is acidified before the addition of the purifying iodine soln., the critical pH range is passed so rapidly that the reaction  $\text{S}^{2-}$  to S is strictly stoichiometric and the uncontrollable oxidation is avoided. Minor procedural modifications are proposed. L. S. ADLER

**1181. Amperometric determination of sulphate ions.** V. V. Ten'kovtsev (Sci. Res. Accumulator Inst., Leningrad). *Zhur. Anal. Khim.*, 1957, **12** (4), 504-508.—The soln. (25 ml) is acidified with 1 ml of dil. HCl (1:1) and is treated at 100° with a known vol. of 0.07 N BaCl<sub>2</sub> to precipitate BaSO<sub>4</sub>. The cooled soln. is neutralised to methyl orange and 5 ml of 5 N ammonium acetate is added to give a pH of 5.5 to 6.2. The excess of BaCl<sub>2</sub> is titrated amperometrically at zero potential with 0.07 M K<sub>2</sub>CrO<sub>4</sub>. The method is suitable for determining SO<sub>4</sub><sup>2-</sup> in nickel hydroxide (the sample is dissolved in the minimum amount of HCl) and the active masses of lead accumulators (Pb is pptd. from the soln. of the sample in HCl by means of Zn, the presence of which in soln. does not affect the subsequent determination of SO<sub>4</sub><sup>2-</sup>). The accuracy of the method depends on the smallness of the excess of BaCl<sub>2</sub>. A double titration is recommended for improving the accuracy. For this, the neutralised soln. is treated with ammonium acetate and titrated amperometrically, with two burettes, one containing BaCl<sub>2</sub> soln. and the other K<sub>2</sub>CrO<sub>4</sub> soln. After each addition of BaCl<sub>2</sub> soln. a few drops of K<sub>2</sub>CrO<sub>4</sub> soln. are added. At the end-point the current no longer increases when the K<sub>2</sub>CrO<sub>4</sub> is added. The small excess of BaCl<sub>2</sub> is now titrated with K<sub>2</sub>CrO<sub>4</sub> from the same burette after addition of 5 ml of ethanol to the soln. The method can be improved further by applying a small correction factor ( $\approx 3\%$ ) to allow for co-pptn. of alkali and heavy metals, as it is found that the amount pptd. is approx. proportional to the wt. of BaSO<sub>4</sub>. G. S. SMITH

**1182. Titrimetric determination of sulphamic acid.** C. L. Whitman (U.S. Naval Powder Factory, Indian Head, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1684-1685.—The method described for the determination of sulphamic acid (in concn. up to 0.001 M) or of HNO<sub>2</sub> in the presence of HNO<sub>3</sub> is based on the interaction of sulphamic acid with NaNO<sub>2</sub>, oxidation of the excess of nitrite with Ce<sup>4+</sup> and titration of unchanged Ce<sup>4+</sup> with ferrous ammonium sulphate. The qualitative differentiation between sulphamic and nitrous acids is achieved by running two blank titrations. G. S. ROBERTS

**1183. Detection of selenium by means of N-phenylanthranilic acid.** V. V. Patsuk (V. V. Vakhurshev Sverdlovsk Mining Inst.). *Zhur. Anal. Khim.*, 1957, **12** (4), 509-512.—With contents of Se  $\leq 5.0$  mg per ml, one drop of the soln. is added to 1 ml of a mixture of 2 vol. of pure H<sub>2</sub>SO<sub>4</sub> not containing oxidising or reducing agents and 1 vol. of water and one drop of a 1% soln. of N-phenylanthranilic acid in conc. H<sub>2</sub>SO<sub>4</sub>, and the soln. is heated nearly to boiling. A blue-violet colour appears. With lower contents, a mixture of 0.5 ml of the soln. and 1 ml of conc. H<sub>2</sub>SO<sub>4</sub> is treated with one drop of the reagent soln. and heated nearly to boiling. By this means,  $5 \times 10^{-7}$  g of Se in the form SeO<sub>4</sub><sup>2-</sup> or  $3 \times 10^{-6}$  g of Se in the form SeO<sub>3</sub><sup>2-</sup> can be detected at a dilution of 1 in  $1 \times 10^5$ . Interference is caused by SnCl<sub>4</sub>, FeCl<sub>2</sub>, FeCl<sub>3</sub> and other oxidising and reducing agents. No interference is caused by Sb<sup>3+</sup>, As<sup>3+</sup>, Sn<sup>4+</sup>, Mo, Cu, Al, Zn and Ti. The colour intensity of the soln. obeys Beer's law. G. S. SMITH

**1184. Determination of molybdenum in uranium-molybdenum alloys by monochromatic X-ray absorption.** W. C. Dietrich and R. E. Barringer (Union Carbide Nuclear Co., Y-12 Plant, Oak Ridge, Tenn.). *U.S. Atomic Energy Comm.*,

Rep. Y-1153, 1957, 14 pp.—A rapid method is described for the determination of 2 to 5% of Mo. The method consists of a monochromatic X-ray absorption analysis for Mo after the major portion of U has been removed by a tributyl phosphate extraction. The limit of error for a single determination at a 95% confidence level is  $\pm 2.7\%$  of the Mo present. NUCL. SCI. ABSTR.

**1185. Separation of molybdenum from iron, aluminium and calcium by means of anionites.** Yu. V. Morachevskii and M. N. Gordeeva (Leningrad State Univ.). *Zavod. Lab.*, 1957, **23** (9), 1066-1067.—Separation of Mo from Fe, Al and Ca in N HNO<sub>3</sub> or dil. HCl soln. is carried out by means of a column of anion-exchange resin PE-9 which has been treated with N HCl and then with 0.5 N NaOH followed by N HNO<sub>3</sub> or dil. HCl to convert it to the nitrate or chloride form. The Fe, Al and Ca pass through, and the Mo on the column is extracted by means of 5% NaOH soln. Retention of the Mo is nearly complete (97 to 98% of the amount present); at higher acidities the separation is less favourable. G. S. SMITH

**1186. Separation of uranium(VI) by paper chromatography.** G. Almássy and M. Vigvári (Inst. Exp. Physics, L. Kossuth Univ., Debrecen). *Acta Chim. Acad. Sci. Hung.*, 1957, **11** (1-2), 1-6 (in English).—Uranium can be separated from all other metal ions by ascending paper chromatography with a solvent mixture of ether (70 ml), ethanol (23 ml) and HNO<sub>3</sub> (7 ml) and the spot can be detected by treatment with K<sub>4</sub>Fe(CN)<sub>6</sub>. The limit of detection is 1  $\mu$ g of U. By comparison with a reference chromatogram, U can be determined with an error of  $\pm 30\%$ . G. BURGER

**1187. Improvements in the fluorimetric determination of uranium.** F. A. Centanni and T. J. Morrison, jun. (Nat. Lead Co., Inc., Raw Mat. Dev. Lab., Winchester, Mass.). *U.S. Atomic Energy Comm.*, Rep. WIN-63, 1957, 22 pp.—Modifications in the instrumentation and method used in the fluorimetric determination of U are reported. The elimination of the reference source in the Galvanek-Morrison fluorimeter has improved the operating stability of the instrument and simplified its operation. Also reported is an automatic fusion burner unit, which minimises the atmosphere-temperature fluctuations and makes possible better reproducibility and greater accuracy in the uranium determination. NUCL. SCI. ABSTR.

**1188. Determination of uranium in solution by X-ray spectrometry.** H. M. Wilson and G. V. Wheeler (Phillips Petroleum Co., Atomic Energy Div., Idaho Falls). *U.S. Atomic Energy Comm.*, Rep. IDO-14393, 1957, 19 pp.—A rapid precise method for the determination of U in HNO<sub>3</sub> soln. is presented. The U in soln. is excited by a primary X-ray beam from a molybdenum tube. The intensity of the fluorescent U L $\alpha$  line is measured by means of a crystal spectrometer, with a scintillation counter and pulse-height analyser. This intensity is converted to concn. by reference to standard soln. The modifications of commercial equipment that simplified the soln. analysis are described, as are comparisons of various instrument components. NUCL. SCI. ABSTR.

**1189. Fluorescence of uranyl salts in solution.** M. Novák (Inst. Nucl. Physics, Acad. Sci., Prague, Czechoslovakia). *Jaderná Energie*, 1957, **3** (2), 44-47.—The bluish-green fluorescence of soln. of

uranyl salts in conc.  $\text{H}_2\text{SO}_4$  can be used for a rapid determination of U. When using the Pulfrich photometer for the measurement of the fluorescence, the error is  $> 10\%$ . The method is suitable only for pure soln. of uranyl ions, because in the presence of other ions in concn.  $> 6 \times 10^{-6}$  g per litre the fluorescence disappears. J. ZYKA

**1190. Uranium determination by the isotope dilution technique.** P. Goris, W. E. Duffy and F. H. Tingey (Phillips Petroleum Co., Idaho Falls, U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1590-1592.—The total uranium from 0.001 to over 350 mg per ml and the uranium isotope distribution can be determined in complex solutions of spent reactor fuel elements. The sample is spiked with  $50 \mu\text{g}$  of  $^{233}\text{U}$  and measurements are made by surface-ionisation mass spectrometry. The error for a single determination is  $\pm 3.7\%$  for the total uranium and  $\pm 0.13\%$  for the major isotope ( $P = 0.95$ ). K. A. PROCTOR

**1191. Beta-gamma counting method for radio-metric determination of uranium.** M. C. Greaves (Territory Enterprises Pty. Ltd., Rum Jungle, Northern Territory, Australia). *Proc. Aust. Inst. Min. Metall.*, 1957, (182), 23-36.—An account is given of the theory and practice of determining the  $\text{U}_3\text{O}_8$  content of ores by determination, separately, of the  $\beta$ - and  $\gamma$ -activities. S.C.I. ABSTR.

**1192. Determination of the oxygen-uranium atomic ratio in non-stoichiometric uranium dioxide and other oxides of uranium.** N. F. H. Bright, L. G. Ripley, J. F. Rowland and R. H. Lake (Dept. Mines and Techn. Surveys, Mines Branch, Canada). *U.S. Atomic Energy Comm., Rep. MD-207*, 1956, 37 pp.—Three methods of determining the O to U atomic ratio in uranium oxides from the stoichiometric compound  $\text{UO}_2$ , up to non-stoichiometric oxides having the formula  $\text{UO}_{2-x}$ , and also in certain higher oxides of U have been evaluated. NUCL. SCI. ABSTR.

**1193. Uranyl and thorium selenites.** E. I. Krýlov and V. G. Chukhlantsev (S. M. Kirov Ural Polytech. Inst.). *Zhur. Anal. Khim.*, 1957, **12** (4), 451-456.—The pH values corresponding to the appearance of opalescence and complete pptn. for pptn. in both  $\text{HNO}_3$  and  $\text{H}_2\text{SO}_4$  soln. of the selenites of  $\text{UO}_2^{2+}$  and  $\text{Th}^{4+}$  are determined. The solubility products at  $20^\circ$  are  $3.8 \times 10^{-11}$  for  $[\text{UO}_2^{2+}][\text{SeO}_3^{2-}]$  and  $1.35 \times 10^{-20}$  for  $[\text{Th}^{4+}][\text{SeO}_3^{2-}]^2$ . G. S. SMITH

**1194. Determination of uranium in a mixture of bromine, bromine trifluoride and uranium hexafluoride.** W. J. Gardner (N. Amer. Aviation, Inc., Downey, Calif.). *U.S. Atomic Energy Comm., Rep. NAA-SR-237*, 1957, 10 pp.—In the method described, the  $\text{UF}_6$  is distilled over together with some Br and  $\text{BrF}_3$  in to a bed of crystalline  $\text{H}_3\text{BO}_3$ , where it is absorbed. The  $\text{H}_3\text{BO}_3$  reaction mixture is heated after the addition of  $\text{H}_2\text{SO}_4$  and excess of HF. Volatile fluorine compounds and boron compounds are driven off to leave a dry residue of  $\text{UO}_2\text{SO}_4$ . Uranium in the  $\text{UO}_2\text{SO}_4$  is determined by standard methods. NUCL. SCI. ABSTR.

**1195. Separation of uranium and vanadium by means of anionite resins.** Yu. V. Morachevskii and M. N. Gordeeva. *Vestn. Leningr. Univ.*, 1957, No. 10 (Part 2), 148-151.—On a column of PE-9 or EDÉ-10, uranium is completely adsorbed from

soln. of  $\text{HCl} > 8 N$ . No adsorption takes place from soln. 0.05 to 4 N in  $\text{HCl}$ , and U that has been adsorbed can be washed through by means of water. No adsorption of V takes place at any acidity, although the vol. of liquid required to wash a column free from V increases with the acid concn. In 8 N  $\text{HCl}$ , U and V can be separated by means of the column. With 0.5 to 12 mg of U and 0.25 to 48 mg of V the error in the determination of either element is  $< 2\%$ . G. S. SMITH

**1196. Separation of small amounts of quadrivalent uranium by the phosphate method in the presence of niobium and tantalum.** Yu. V. Morachevskii and I. A. Tserkovnitskaya. *Vestn. Leningr. Univ.*, 1957, No. 10 (Part 2), 152-154.—The soln. containing  $> 0.5$  g of tartaric acid in 100 ml is reduced with amalgamated zinc, 1 ml of a 2% soln. of  $\text{Zr}(\text{NO}_3)_4$  is added as a collector, and U together with Zr is pptd. by addition of 10%  $(\text{NH}_4)_2\text{HPO}_4$  soln. After 1 hr. in the cold, the ppt. is collected and washed with a soln. prepared by mixing 50 ml of N sodium acetate, 70 ml of N  $\text{HCl}$  and 130 ml of water. The ppt. is then dissolved in 10 N  $\text{H}_2\text{SO}_4$  and the U is titrated with 0.01 N ammonium vanadate soln. in the presence of N-phenylanthranilic acid as indicator. With higher concn. of tartaric acid or with oxalic acid even of low concn., compounds of  $\text{UO}_2$  are only partially reduced or not at all. Reduction of  $\text{Nb}^{5+}$  takes place partially under the conditions for reducing  $\text{UO}_2$  but re-oxidation occurs rapidly when the soln. stands in contact with air. The method is suitable for determining U in the presence of Nb and Ta. G. S. SMITH

**1197. Deposition of uranium by the method of internal electrolysis in the presence of vanadium, chromium, nickel and cobalt.** Yu. V. Morachevskii and I. A. Tserkovnitskaya. *Vestn. Leningr. Univ.*, 1957, No. 16 (Part 3), 127-130.—With a zinc (or cadmium) rod as anode and a platinum-gauze cathode the use of EDTA (disodium salt) as a complex former with V, Al, Ni, Co and Cr permits U to be quantitatively separated by internal electrolysis at pH 5. G. S. SMITH

**1198. Sequential analysis of tracer amounts of neptunium, uranium and plutonium in fission-product mixtures by anion exchange.** L. Wish and M. Rowell (Naval Radiol. Defense Lab., San Francisco). *U.S. Atomic Energy Comm., Rep. USNRDL-TR-117*, 1956, 34 pp.—Elution characteristics of Np, Pu, U and Zr in  $\text{HCl}$ ,  $\text{HNO}_3$  and  $\text{H}_2\text{SO}_4$  media with Dowex-2 anion resin were found. From these a procedure for the sequential analysis of the three actinides in fission-product mixtures in  $\text{HCl}$  and  $\text{HNO}_3$  has been developed. Protactinium has also been included in the separation scheme. In most cases the Np, Pa and Pu were sufficiently decontaminated after one column for 99% purity. The U contained a large portion of the Zr and had to be treated further for a satisfactory separation. The yields were approx. 70 to 90%. NUCL. SCI. ABSTR.

**1199. Determination of total plutonium in the presence of aluminium.** R. E. Burns and G. B. Barton (Hanford Works, Richland, Wash.). *U.S. Atomic Energy Comm., Rep. HW-15944*, 1957, 7 pp.—The adoption of aluminium nitrate as salting agent in the redox process made it imperative that a method be available for determining Pu in the presence of Al. Large amounts of Al have been found to interfere with the determination of Pu by

the lanthanum fluoride procedure. Previous attempts to increase the accuracy of the lanthanum fluoride method, by pptng.  $\text{LaF}_3$  from 4 M HF (rather than 2M), have been successful only when the initial plutonium level was high. A method has been developed which permits recoveries of Pu greater than 96% from 1.5 M aluminium nitrate soln. Quantitative carrying of Pu (mixed oxidation states) on a lanthanum hydroxide-sodium diuranate ppt. serves to separate the Pu from the Al as well as from any dichromate present. The ppt., after being washed with dil. NaOH, is dissolved in 2 M HCl. A standard lanthanum fluoride procedure then follows. An outline of the complete procedure is included. NUCL. SCI. ABSTR.

**1200. Spectrographic estimation of major-constituent plutonium by the iron-flux method.** F. T. Birks. A.E.R.E. Report C/R 2081, 1957, 5 pp.—The amount of Pu retained on an ion-exchange resin after elution was determined by ignition of a portion of the resin to remove the organic matter, and analysing the residue spectrographically by the iron-flux method of Garton *et al.* (CCI, 1950, Rep. No. 8007), with a flux comprising equal parts of anhyd.  $\text{FeSO}_4$  and  $(\text{NH}_4)_2\text{SO}_4$  to provide the buffer matrix from which the added elements are volatilised into the arc. The concn. range covered was 0.5 to 100% of  $\text{PuO}_2$  and at the 50% level the standard deviation of the results was 0.34 and the coeff. of variation was 6.6%. J. M. JACOBS

**1201. Micro-determination of elementary fluorine.** E. A. Perehud and B. S. Bolkina (State Sci. Res. Inst. of Work Hygiene and Occupational Diseases, Leningrad). *Zhur. Anal. Khim.*, 1957, **12** (4), 513-515.—One method is based on the reaction of Br, set free by the action of F on KBr, on fluorescein, which gives a red colour due to eosin. Another method is based on the decolorisation of methyl red soln. by F. The methods when applied to the determination of F in air are sensitive to  $\approx 0.35 \mu\text{g}$  per litre when one litre of air is used. G. S. SMITH

**1202. Qualitative and semi-quantitative detection of the fluorine ion by a drop method.** V. P. Gladyshev and G. A. Tolstikov (Kazakh State Univ., Alma-Ata). *Zhur. Anal. Khim.*, 1957, **12** (4), 567-568.—With contents of F > 1 mg per ml, a drop of 0.1 M  $\text{K}_4\text{Fe}(\text{CN})_6$  is placed on filter-paper, followed by a drop of the soln. to be tested. One drop of 0.1 M  $\text{FeCl}_3$  is then placed in the centre of the spot. In the presence of  $\leq 2$  to  $3 \mu\text{g}$  of F in one drop (0.005 ml), a pink or lilac ring appears. With lower contents of F, two separated drops of 0.005 M  $\text{FeCl}_3$  are placed on filter-paper, and on one is placed a drop of the soln. to be tested. A difference in colour is noticed. The detection of  $0.01 \mu\text{g}$  of F at a limiting dilution of 1 in  $1 \times 10^5$  is possible. G. S. SMITH

**1203. Spectrophotometric determination of inorganic fluoride and of fluorine in organic compounds.** R. P. Curry and M. G. Mellon (Purdue Univ., Lafayette, Ind., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1632-1637.—The fluorine in a sample of an inorganic fluoride is distilled into a sodium borate-boric acid buffer soln. as silicon tetrafluoride, where it hydrolyses to form a soluble silicate. The silicate is then allowed to react with ammonium molybdate soln. in acid medium, and the resulting molybdosilicic acid is treated with 1-amino-2-naphthol-4-sulphonic acid to give a blue heteropoly compound. The colour is measured at  $700 m\mu$  in a Beckman DU

quartz spectrophotometer by the differential method. The fluorine in an organic compound is converted into silicon tetrafluoride by combustion in a tube packed with quartz chips (Freier *et al.*, *Anal. Chem.*, 1955, **27**, 146); the procedure for inorganic fluorides is then followed. This method has been applied to organic fluoro compounds containing Br, Cl, S, O and N, and has given accurate results. The interference effects of 25 different ions likely to be present during the combustion were studied; of these only borate was found to be undesirable. The method is rapid (about 1.5 hr. per determination) and precise (coeff. of variation of  $\pm 0.3\%$  F for 35 determinations). G. S. ROBERTS

**1204. Determination of fluoride ion in cryolite by means of ion-exchange chromatography.** Sh. K. Ashratova (All-Union Sci. Res. Inst. of Glass Fibres). *Zavod. Lab.*, 1957, **23** (9), 1064-1065.—A column of KU-1 or KU-2 resin (H form) contained in a tube 45 cm long and 1.2 cm in internal diameter made of methacrylate resin (shown to be completely unaffected by HF) is used to adsorb Na and Al from cryolite soln. The HF that is formed appears in the filtrate and is titrated with 0.1 N NaOH in the presence of phenol red indicator. G. S. SMITH

**1205. Remote determination of fluoride in zirconium-uranium fuel processing solutions.** F. W. Dykes, G. L. Booman, M. C. Elliott and J. E. Rein (Phillips Petroleum Co., Atomic Energy Div., Idaho Falls). *U.S. Atomic Energy Comm., Rep. IDO-14405*, 1957, 13 pp.—An analytical method and apparatus are described for use with highly radioactive Zr-U fuel recovery samples. Fluoride is separated by pyrohydrolysis in a nickel combustion apparatus. The evolved HF is then titrated with standard  $\text{Th}(\text{NO}_3)_4$  to a conductimetric end-point. Under routine conditions, for sample aliquots containing from 0.25 to 1 milli-equiv. of fluoride, the precision is  $\pm 0.055$  milli-equiv. standard deviation for a single determination. NUCL. SCI. ABSTR.

**1206. Modified zirconium-Eriochrome cyanine R determination of fluoride.** L. L. Thatcher (U.S. Geological Survey, Washington, D.C., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1709-1712.—The methods developed by Lamar (*Ind. Eng. Chem., Anal. Ed.*, 1945, **17**, 48) and Megregian (*Anal. Chem.*, 1954, **26**, 1161) have been investigated and improved. A single, stable reagent soln. has been produced, interference by ions commonly present in natural and polluted waters has been reduced, and errors due to sulphate have been eliminated. The range of the method has been extended. G. S. ROBERTS

**1207. Determination of small amounts of chloride in high nickel-chromium-iron solutions.** K. W. Puphal, G. L. Booman and J. E. Rein (Phillips Petroleum Co., Atomic Energy Div., Idaho Falls). *U.S. Atomic Energy Comm., Rep. IDO-14389*, 1956, 10 pp.—Turbidimetric measurement of colloidal  $\text{AgCl}$  is reliable for the determination of small amounts of  $\text{Cl}^-$  in samples containing high concn. of  $\text{Fe}^{3+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{UO}_2^{2+}$ ,  $\text{HNO}_3$  and HF, and low levels of  $\text{Nb}^{5+}$ . Ethanediol, 50% by vol., is the recommended medium. At the working wavelength of  $475 m\mu$ , the optical absorbance from the diverse ions is a minimum and is linear with  $\text{Cl}^-$  concn. For sample aliquots containing  $5 \times 10^{-3}$  mg of  $\text{Cl}^-$ , the precision is better than 20% standard deviation and is 1.2% at the 0.1-mg level. NUCL. SCI. ABSTR.



1208. Quantitative determination of iodine and iodides with hydrochloric acid solution of iodine trichloride. F. E. Kagan (Kiev Post-grad. Med. Inst.). *Apteknoe Delo*, 1957, 6 (1), 14-17.—Methods are given for the determination of iodine, potassium iodide and mercuric iodide by direct titration against a soln. of  $\text{ICl}_3$  in  $\text{HCl}$ . Potassium iodide can be determined in the presence of  $\text{CaCl}_2$ , theobromine, Na salicylate, caffeine, iodine, petroleum jelly and other ingredients of pharmaceutical preparations. E. HAYES

1209. The permanganate-bromide system in derivative polarography. N. Ya. Khlopin and L. G. Gein (Molotov State Pharm. Inst.). *Zhur. Anal. Khim.*, 1957, 12 (4), 561-563.—The effects of various factors on the reaction between  $\text{KMnO}_4$  and  $\text{Br}^-$  in acid soln. are studied in connection with the polarographic determination of Mn. G. S. SMITH

1210. Potentiometric titration of manganese in ferrous and non-ferrous metals and alloys, in ores, minerals and other materials with micro sample weights. A. S. Slavtinskii (A. A. Balkov Inst. of Metallurgy, USSR, Moscow). *Zhur. Anal. Khim.*, 1957, 12 (4), 485-488.—Titration is carried out with a rotating platinum electrode and 0.001 N  $\text{KMnO}_4$ . A valve potentiometer of special construction is described. The sample (15 to 20  $\mu\text{g}$ ) is weighed on a torsion micro-balance. G. S. SMITH

1211. Paper chromatography of inorganic ions. XVI. A note on the paper chromatography of technetium using a long-life isotope. M. Levi and M. Lederer (Lab. Curie, Inst. du Radium, Paris). *J. Inorg. Nuclear Chem.*, 1957, 4 (5-6), 381-382.—The best separation of Tc and Re with Whatman No. 1 paper and the ascending technique was obtained with butanol-conc.  $\text{HCl}$  (1:1) as solvent. Rhenium and Tc can be separated from W and Mo by using butanol shaken with 10% acetic acid. G. J. HUNTER

1212. Volumetric determination of trivalent iron by means of triphosphoric acid. L. Jankovits (Inst. Gen. Chem., Tech. Univ., Budapest). *Acta Chim. Acad. Sci. Hung.*, 1957, 11 (1-2), 185-194 (in German).—This article has been published in Hungarian, in *Magyar Kém. Foly.*, 1956, 62 (6), 191. Cf. *Anal. Abstr.*, 1957, 4, 498. G. BURGER

1213. Ultra-violet spectrophotometric determination of iron(III) as acetato complex. Masayoshi Ishibashi, Tsunenobu Shigematsu, Yuuroku Yamamoto, Masayuki Tabushi and Minoru Kitayama (Inst. Chem. Res., Kyoto Univ., Japan). *Bull. Inst. Chem. Res., Kyoto Univ.*, 1957, 35 (1-2), 6-15.—Milligram amounts of  $\text{Fe}^{3+}$  can be determined in soln. of ferric perchlorate containing acetic acid ( $< 5 M$ ) by a differential u.v. absorption method. Procedure.—The sample (e.g., 0.15 to 0.2 g of iron alloy) is dissolved in warm 4 M  $\text{HClO}_4$ ; a few drops of 30%  $\text{H}_2\text{O}_2$  are added to the cool soln., which is then evaporated to dryness several times, the final residue being dissolved in  $\text{H}_2\text{O}$  and the soln. made up to 500 ml. To an aliquot (20 ml) are added 50 ml of glacial acetic acid and 30 ml of  $\text{H}_2\text{O}$ , and the extinction of the soln. is measured at 335  $m\mu$  (1-cm silica cell) against a standard soln. of Fe (10 to 50 p.p.m.). Beer's law is valid for concn. of Fe  $> 80$  p.p.m.; the mean error is  $\approx \pm 0.4\%$ , but is much higher for Fe-V alloys. Interference by Cu is almost negligible, but  $> 10$  p.p.m.

of  $\text{Ti}^{4+}$  should be present. Measurements can be made between  $10^\circ$  and  $35^\circ$  without any temp. effect. W. J. BAKER

1214. Spectrophotometric investigations on the iron(III)-morellin complex. S. G. Tandon and C. C. Patel (Indian Inst. Sci., Bangalore). *J. Indian Inst. Sci.*, 1957, 39 (2), 129-137.—Spectrophotometric investigations on soln. of  $\text{FeCl}_3$  and  $\text{Fe}^{III}$ -morellin complex in an absolute ethanol medium show that there is a continuous shift in the absorption max. of  $\text{FeCl}_3$  from 410  $m\mu$  to 490  $m\mu$  as the concn. of  $\text{FeCl}_3$  is raised from  $2.24 \times 10^{-4}$  to  $2.30 \times 10^{-3} M$ . One complex of the molar composition  $\text{Fe}_2\text{R}_3$  ( $R = \text{morellin}$ ), with an absorption max. at 550  $m\mu$ , is formed when  $\text{FeCl}_3$  is mixed with morellin. This complex formation can be used to determine  $\text{Fe}^{III}$  in the range 0.5 to 19 p.p.m. The complex dissociates in the absolute ethanol medium with an equilibrium constant (determined spectrophotometrically) averaging  $4.95 \times 10^{-17}$  at  $25^\circ$ , and has a free energy of formation of  $-22.15$  kcal. per mole. I. JONES

1215. Chemical analysis and separation by extraction methods. V. Separation of iron from vanadium. Sakujiro Yamamoto (Fac. of Liberal Arts, Shimane Univ., Matsue). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1957, 78 (6), 888-896.—The extraction of Fe ( $< 0.1$  g) from 6 N  $\text{HCl}$  is increasingly inhibited by V (metavanadate) with an increasing amount of V (5 to 150 mg). The use of a mixture of diethyl ether and ethyl acetate (4 + 1) markedly decreases the interference, so that  $< 0.1$  g of Fe is quant. collected from 6 N  $\text{HCl}$  soln. in the presence of 25 mg of V, by three extractions with this mixture. A significant amount of V is also extracted. A similar trend was observed by the use of diisopropyl ether in place of diethyl ether. The titration method with  $\text{Na}_2\text{S}_2\text{O}_8$  (*Ibid.*, 1953, 74, 941) is applicable in the presence of V. Vanadium ( $> 0.1 \mu\text{g}$ ) may be detected by extraction with diethyl ether from an acid soln. containing KSCN and phenylhydrazine when it gives a violet soln.; Fe ( $> 0.1 \mu\text{g}$ ) is also extracted under these conditions to give a brown coloration, but this disappears on treatment with 7 N  $\text{HCl}$ . K. SAITO

1216. Volumetric determination of iron, platinum and iridium when present together. A. A. Grinberg and A. I. Dobroborskaya. *Zhur. Neorg. Khim.*, 1956, 1 (10), 2360-2367; *Ref. Zhur., Khim.*, 1957, Abstr. No. 30,924.—The potentiometric titration of  $\text{Fe}^{2+}$  and  $[\text{PtCl}_4]^{2-}$  with  $\text{Ce}(\text{SO}_4)_2$  has been extended to the analysis of a soln. containing  $(\text{NH}_4)_2[\text{IrCl}_6]$ ,  $\text{K}_2[\text{PtCl}_6]$  and  $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ . The  $\text{Fe}^{2+}$  are determined by titration with  $\text{Ce}(\text{SO}_4)_2$  in the presence of  $\text{H}_2\text{SO}_4$ ,  $\text{NaH}_2\text{PO}_4$  and diphenylamine; the sum of  $\text{Fe}^{2+}$ ,  $\text{Ir}^{3+}$  and  $\text{Pt}^{2+}$  is determined by potentiometric titration with  $\text{KMnO}_4$ , whence the sum of  $\text{Ir}^{3+}$  and  $\text{Pt}^{2+}$  can be determined by difference. C. D. KOPKIN

1217. Quantitative spectrochemical determination of iron and titanium in kaolin with direct and alternating current arcs. F. Hegemann, H. Kostyra and G. Wilk (Inst. der Tech. Hochschule, Munich). *Ber. dtsh. keram. Ges.*, 1957, 34 (4), 87-92.—Full details are given of the application of a Zeiss Q.24 spectrograph to the calcined sample powder with Ni as the internal standard (3050-819 Å) in a hole in the anode of a d.c. carbon arc. By using Ti 3088-025 Å and 3234-516 Å and Fe 3047-605 Å and 3020-640 Å the coeff. of variation for results for

$\text{Fe}_2\text{O}_3$  and  $\text{TiO}_2$  contents of  $\approx 0.3\%$  were, respectively,  $\pm 2.8$  and  $4.1\%$ . The raw kaolin gave less accurate results. With a pelleted calcined sample in an a.c. (condenser) arc (Fe 3020-640 Å and Ti 3241-986 Å) the coeff. of variation was  $\pm 1.7\%$  for each oxide. J. A. SUGDEN

**1218. Use of acetylacetone extractions in ferrous analysis.** J. P. McKaveney (Univ. Pittsburgh, Pa.). *Dissert. Abstr.*, 1957, **17** (5), 971.—Extraction data were obtained by the method described for the extraction of the ions  $\text{Cr}^{3+}$ ,  $\text{Fe}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Mo}^{6+}$ ,  $\text{Ti}^{4+}$ ,  $\text{V}^{3+}$ ,  $\text{V}^{4+}$  and  $\text{W}^{6+}$  by acetylacetone. Analysis procedures, particularly for determinations of extremely low concentrations, were developed for Cr, Mo and V in ferrous materials.

O. M. WHITTON

**1219. Polarographic analysis of iron ores.** M. L. Borlera (Turin Polytech., Italy). *Ric. Sci.*, 1957, **27** (5), 1492-1499.—A rapid method has been developed for the determination of Cu, Mn, Pb, As, S and Zn in iron ores or roasted ores such as pyrites. To overcome the interference of As in the determination of Pb, the As is removed by volatilisation as  $\text{AsCl}_3$ , and interference from  $\text{SO}_4^{2-}$  is avoided by adding KCl to increase the solubility of  $\text{PbSO}_4$ . The procedure is particularly suitable for small amounts of As; 20  $\mu\text{g}$  or 0.004% may be determined. L. A. O'NEILL

**1220. Some recent advances in the analysis of cast iron and foundry materials.** W. E. Clarke (B.C.I.R.A., Alvechurch, Birmingham). *Metalurgia, Manch.*, 1957, **56**, 47-52.—Recent work carried out by the Methods of Analysis Subcommittee of the B.C.I.R.A. is reviewed. Among the subjects discussed are—sampling; use of high-frequency heating in determination of C by the combustion method; rapid methods for the determination of Mg; determination of trace elements (Sb, Pb, Bi) in cast iron, and of Al in cupola slags and cast irons. (19 references.) S.C.I. ABSTR.

**1221. Determination of aluminium in cast iron and ferrosilicon by the fluoride volumetric method.** W. E. Clarke and R. C. Rooney (B.C.I.R.A., Alvechurch, Birmingham). *J. Res. Brit. Cast Iron Ass.*, 1957, **6** (12), 666-669.—The technique applied to cupola slags (cf. Clarke, *Anal. Abstr.*, 1957, **4**, 53) has now been successfully applied to cast iron and ferrosilicon. After the sample has been dissolved by appropriate methods, the major part of the Fe is removed by a butyl acetate separation and other interfering elements by a diethyldithiocarbamate- $\text{CHCl}_3$  extraction. The pH of the soln. is then adjusted and neutral aq. KF added, the liberated  $\text{OH}^-$ , corresponding to the Al present, being titrated with 0.1 N HCl. S.C.I. ABSTR.

**1222. Photometric determination of aluminium in steel by means of Eriochrome cyanine.** H. Lilie (Dtsh. Amt. für Material- und Warenprüfung, Halle, Germany). *Chem. Tech., Berlin*, 1957, **9** (6), 364.—Steel is treated with  $\text{H}_2\text{SO}_4$  and small amounts of  $\text{HNO}_3$  until a clear soln. is obtained. The soln. is evaporated to dryness and the  $\text{HNO}_3$  is driven off. Water and HCl are added and the soln. is filtered, then treated with thioglycolic acid and carefully neutralised with NaOH, to reduce the Fe. An aq. soln. of Eriochrome cyanine is added which forms a red lake with the Al present. The simultaneous colour formation due to the interaction between the dyestuff and thioglycolic acid

is compensated by making a blank test with pure Fe. The method is suitable for contents of Al up to 2%. The accuracy is not stated. C. A. CURTIS

**1223. Spectrochemical determination of silicon in low or high alloy steels.** H. Carrancio de la Plaza. *Inst. Hierro y Acero*, 1957, **10** (52), 195-199.—Silicon in high and low alloy steels is determined by comparing the intensities of two lines of the same element, eliminating an internal standard. The data are compared with results from the Gerlach method of comparing the intensity of a line of the element to be determined with a line of a standard element. Experimental details are given for eight Spanish steels. Line densities were measured on photographic plates with a Hilger microphotometer, with a slit 15 mm long and 0.2 mm wide, and the pairs of lines chosen were those nearest and closest in intensity. Results were good for concn. between 0.1 and 2.85%. R. L. MORTLOCK

**1224. Colorimetric method for the determination of phosphorus in steel.** F. Burriel-Martí, V. Hernando Fernández and J. Rodríguez Señas (Dept. of Anal. Chem., C.S.I.C., Madrid, Spain). *An. Real Soc. Esp. Fis. Quim.*, B, 1957, **53** (5), 361-368.—A previously reported method (Burriel-Martí and Hernando Fernández, *An. Inst. Esp. Edafol. Fisiol. Veg.*, 1947, **6**, 543) for the determination of P in soil is modified (to avoid interference of a strong concn. of Fe) for the determination of P in steel. Blanks are necessary to allow for the effect of other alloy metals (Cr, W, Mo, V, Ni, etc.). The sample is dissolved in  $\text{HCl-HNO}_3\text{-HClO}_4\text{-H}_2\text{SO}_4$  (5:5:5:2), converted into the molybdenum blue complex, and determined absorptiometrically, with a red filter (Ilford 608) and the use of calibration curves. C. A. FISCH

**1225. Photometric determination of boron in steel.** I. U. Martynchenko and A. M. Bondarenko (A. M. Gorki Kharkov State Univ.). *Zhur. Anal. Khim.*, 1957, **12** (4), 495-498.—The method is based on the use of carmine. The sample (1 g) of steel in a quartz flask fitted with a reflux condenser and a delivery funnel is treated with 10 ml of dil.  $\text{H}_2\text{SO}_4$  (1:4), 4 ml of 30%  $\text{H}_2\text{O}_2$  soln. is added dropwise and the boiling is continued until the carbides are dissolved. Then 2.5 ml of conc.  $\text{H}_3\text{PO}_4$  is added and boiling is continued for 20 to 30 min. to destroy  $\text{H}_2\text{O}_2$ . The cooled soln., together with water washings of the condenser, is treated dropwise in slight excess with 0.1 N  $\text{KMnO}_4$ , the excess is destroyed by addition of 0.1 N  $\text{FeSO}_4$ , and the soln. after dilution to 25 ml is filtered through a dry filter. An aliquot (2.5 ml) is mixed with 10 ml of conc.  $\text{H}_2\text{SO}_4$  and quickly cooled, 10 ml of a 0.05% soln. of carmine in conc.  $\text{H}_2\text{SO}_4$  is added and the soln. is diluted to 25 ml with conc.  $\text{H}_2\text{SO}_4$ . The extinction is measured with a green filter after 1 hr. A blank is carried out with a similar steel free from B or a B-free plain carbon steel to which soln. corresponding to the alloying elements are added. G. S. SMITH

**1226. Colorimetric analysis by photo-electric method: determination of phosphorus, tungsten, silicon, nickel and boron in steel.** E. Piper and H. Hagedorn. *Arch. Eisenhüttenw.*, 1957, **28**, 373-377.—Results from an industrial laboratory are reported. Details are given for the determination of (i) P, by the use of the molybdenum blue reaction; (ii) W, with or without V, by reduction with  $\text{SnCl}_2$  soln. followed by treatment with KSCN soln. to give

a characteristic colour; (iii) Si, by the molybdosilicic acid reaction; (iv) Ni, by the formation of a red soln. with dimethylglyoxime in the presence of Br; (v) B, by the production of a characteristic blue-red lake with carminic acid. E. STERN

**1227. Determination of uranium dioxide in stainless steels by the X-ray fluorescence method.** L. Silverman, W. W. Houk and L. A. Moudy (Atomics Internat. Div., N. Amer. Aviation, Inc., Canoga Park, Calif.). *U.S. Atomic Energy Comm., Rep. NAA-SR-1848*, 1957, 11 pp.—A rapid method for the determination of  $UO_2$  by direct X-ray fluorescent analysis after chemical solution of the sample in  $HClO_4$  is described. Strontium is used as an internal standard. X-ray fluorescence methods of analysis have many advantages over the commonly used chemical methods such as the colorimetric, gravimetric and volumetric, because elements which ordinarily interfere need not be separated before analysis. Statistically, 11 independent soln. were counted and 9 sets of results were obtained on each one. Standard deviations are shown.

NUCL. SCI. ABSTR.

**1228. Detection of cobalt.** K. N. Bagdasarov, P. N. Kovalenko and S. S. Mel'nikova (Rostov-on-Don State Univ.). *Zhur. Anal. Khim.*, 1957, **12** (4), 564-565.—The colour reactions for Co described by Isakov (*Brit. Abstr. C*, 1952, 88) and by Goldberg (*Anal. Abstr.*, 1954, **I**, 2097) are due merely to the effect of dehydrating the compound of Co.

G. S. SMITH

**1229. Photometric determination of cobalt with pyrazolone derivatives.** E. V. Sokolova, A. S. Pesis and N. I. Panova (Molotov Medicinal Inst.). *Zhur. Anal. Khim.*, 1957, **12** (4), 489-494.—The use of methyl, propyl, phenyl, *p*-tolyl, *o*- and *p*-hydroxyphenyl, and *p*-dimethylaminophenyl derivatives of diantipyrinylmethane for colorimetric and photometric determinations of Co is studied. Methods for the determination of Co in bronze and cast iron are given. With bronze, 0.1 g is dissolved in 2 ml of dil.  $HNO_3$ , the soln. is diluted to 100 ml with water, and a 2-ml aliquot containing  $\approx 0.1$  mg of Co is neutralised with N aq.  $NH_3$  and treated with 1 ml of N  $Na_2S_2O_3$  (to react with Fe and Cu), 0.5 ml of saturated  $NH_4SCN$  soln., 1 to 2 ml of a 1% soln. of one of the reagents, 1 ml of 0.1 N  $H_2SO_4$ , and 2 ml of  $CHCl_3$ . The extinction of the blue extract in  $CHCl_3$  is measured.

G. S. SMITH

**1230. Photometric method of determining nickel with dimethylglyoxime.** Z. Gregorowicz, S. Grochowski and J. Kubala (Silesian Polytech., Gliwice, Poland). *Chem. Anal., Warsaw*, 1957, **2** (4), 322-326.—Some results are given on the effect of the relative amount of dimethylglyoxime, and the volume of ethanol in which this is dissolved, on the extinction value of the coloured soln. The optimum is shown to be 1 ml of a 0.1% ethanolic soln. of dimethylglyoxime for amounts of nickel in the range 0.01 to 0.1 mg in a total volume of 50 ml. Under these conditions the extinction is stated to be unchanged after 20 hr. T. M. M.

**1231. Studies on o-dioximes and their metal complexes. VI. Colorimetric determination of nickel with dimethylglyoxime.** Ken-ichiro

Yamasaki and Chuya Matsumoto (Chem. Dept., Kita College, Osaka Univ., Shibahara, Toyonaka). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1957, **78** (6), 833-836.—Nickel yields two complexes with dimethylglyoxime (**I**) (*Ibid.*, 1956, **77**, 1111) having

molar ratios of Ni to **I** of 1:2 and 1:3. The latter complex (max. absorption at 480 m $\mu$ ) is more stable and can be satisfactorily used in the colorimetric determination of Ni. Its formation is favoured by a large excess of **I** and elevated pH. A large excess of Br must be avoided. The presence of Fe (< 230 times the weight of the Ni), Cr (< 3 times), Mn (< 2 times), Cu (< 2 times) and Co (< 2.5 times) does not cause interference with the determination.

**VII. Studies on some ferrous complexes of substituted phenylglyoximes.** Ken-ichiro Yamasaki and Chuya Matsumoto. *Ibid.*, 1957, **78** (6), 837-840.—The ferrous complex of phenylglyoxime (**II**) formed in a neutral soln. (optimum pH 7.0) containing pyridine (**III**) and Na tartrate comprises two mol. each of co-ordinated **II** and **III**. This was confirmed by the preparation of a crystalline compound having max. absorption at 555 m $\mu$ . The introduction of Br or  $-OCH_3$  into the phenyl ring has only a slight effect on the light absorption in  $CHCl_3$ . The extinction coeff. in  $CHCl_3$  at 555 m $\mu$  decreases with time and that at 440 m $\mu$  increases, whilst in  $CHCl_3$  containing **III** they remain unchanged. It is likely that the co-ordinated **III** leaves the ligand in  $CHCl_3$  free from **III**. An orange  $Fe^{II}$  complex (max. absorption 490 m $\mu$ ) is also formed in the absence of **III** under similar conditions, but its extinction coeff. is much smaller.

K. SAITO

**1232. Detection of nickel in the presence of cobalt, copper and other elements.** A. D. Shun'ko (Kharkov Inst. of Soviet Commerce). *Zhur. Anal. Khim.*, 1957, **12** (4), 569.—Into a test-tube are introduced 8 or 9 drops of the soln. to be tested, 7 or 8 times as much saturated ammonium carbonate soln. and 0.5 ml of a suspension prepared by shaking together 2 g of dimethylglyoxime and 30 ml of  $CCl_4$ . After shaking, the lower layer will be coloured pink in the presence of Ni. Detection of Ni is possible in the presence of 500 times as much Co and 1000 times as much Cu. The minimum amount of Ni detectable is 1.8  $\mu$ g, the limiting dilution is 1 in  $2 \times 10^4$ , and the most favourable pH is between 8 and 9.

G. S. SMITH

**1233. Naphthylaminesulphonic acids. A new class of organic reagents for spectrophotometric determination of trace amounts of osmium.** E. L. Steele and J. H. Yoe (Univ. of Virginia, Charlottesville, U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1622-1624.—Twenty-eight naphthylaminesulphonic acids were investigated as colorimetric reagents for Os. All the acid-Os complexes showed a fairly sharp peak at  $\approx 560$  m $\mu$  and all adhered to Beer's law for osmate concn. of 0.1 to 6 p.p.m. Several of the acids have sensitivities as high as 1 part in  $2 \times 10^7$ . Ions of the following metals interfered in  $> 1:1$  concn.— $Ru^{III}$ ,  $Rh^{III}$ ,  $Pd^{II}$ ,  $Ir^{IV}$ ,  $Pt^{IV}$ ,  $Al^{III}$ ,  $Cr^{III}$ ,  $VO^{2+}$ ,  $Fe^{II}$  and  $Fe^{III}$ ,  $Co^{II}$ ,  $Ni^{II}$ ,  $Cu^{II}$ ,  $Zn^{II}$ ,  $Pb^{II}$ ,  $Mn^{II}$ ,  $Mg^{II}$  and  $Ag^+$ ; the Os must be separated from these before determination and this is best achieved by distillation of the tetroxide. Investigation was also made into the structural analysis of the complexes, and the nature, position and number of amine and sulphonic acid groups were studied.

G. P. COOK

**1234. Chemical analysis of traces of platinum in 7% uranium-zirconium.** V. F. Consalvo and J. Rynasiewicz (Knolls Atomic Power Lab., Schenectady, N.Y.). *U.S. Atomic Energy Comm., Rep. KAPL-M-JR-9*, 1957, 6 pp.—A procedure has been

developed for the determination of Pt in a 7% U-Zr alloy containing approx. 0.30% of Pt. Fine metal chips from the alloy were dissolved in HF acid and  $H_2O$ . Nitric acid was added to complete the dissolution of the Zr and U. The acid-insoluble Pt was ashed and then dissolved in aqua regia. The soln. was evaporated to dryness, filtered, and dissolved in HCl. The Pt was determined colorimetrically by the  $SnCl_2$  method.

NUCL. SCI. ABSTR.

**1235. Chemical analysis of binary alloys of platinum and uranium.** M. S. Richmond, J. R. Baldwin and E. J. Maienthal (Nat. Bur. Stand., Washington, D.C.). *U.S. Atomic Energy Comm., Rep. NBS-4555*, 1956, 19 pp.—The analyses of 30 Pt-U alloys containing from 0.2 to 95% of Pt are described. Detailed procedures and experimental data are included. Platinum is determined by a stannous chloride spectrophotometric method in the presence of U or by a sulphide gravimetric method. Provision is made for the separation and/or determination of Cu, W and U that might contaminate the  $PtS_2$  ppt. After the separation of Pt, U is determined by an alkaline peroxide spectrophotometric method, or by titration with dichromate.

NUCL. SCI. ABSTR.

**1236. Infra-red spectra of some heteropoly acid salts.** N. E. Sharpless and J. S. Munday (U.S. Dept. of Health, Educ. and Welfare, Bethesda, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1619-1622.—The i.r. spectra of a group of 12 heteropoly acids and their ammonium salts were investigated in the region 1200 to 625  $cm^{-1}$ . An equation was derived which enables integrated molar extinction coefficients to be calculated for compounds suspended in potassium bromide discs, and these coefficients have been calculated for the compounds investigated and some tentative assignments have been made for various bands.

G. P. COOK

**1237. Examination of Orr and Bankston stearic acid method for determining surface area of clays.** K. S. Birrell (Soil Bureau, D.S.I.R., Auckland, N. Zealand). *N. Z. J. Sci. Tech.*, 1957, **38** (6), 588-594.—A rapid liquid-phase method proposed by Orr and Bankston (*Brit. Abstr. C*, 1952, 250) was tested with a New Zealand kaolin, and with subsoils of moderate to very high surface area. Values representing only a small fraction of the surface as indicated by low-temp. gas adsorption were obtained in all cases. Tests with two forms of amorphous  $Al_2O_3$  indicate that only natural materials in which the particles are coated with an active form of the  $Al_2O_3$  would give approx. correct results.

R. H. HURST

**1238. Methods for the analysis of glass. II. Recommended procedure for the analysis of soda-boric oxide-alumina-silica glasses of high silica and boric acid content.** British Standards Institution (2 Park St., London). B.S. 2649: Part 2: 1957, 20 pp.—For the chemical analysis of borosilicate glasses of the type employed for the manufacture of laboratory apparatus, recommended procedures are described for the determination of silica, aluminium oxide, calcium oxide, sodium oxide, potassium oxide, boric oxide, loss on ignition at 550°, total iron by the thioglycolic acid method, and titanium dioxide, and an alternative procedure for the determination of aluminium oxide and calcium oxide when the determination of silica is not required.

O. M. WHITTON

**1239. Analysis of inorganic constituents of coal by photometric methods.** W. J. S. Fringle (Nat. Coal Bd., Coal Survey Lab., Birmingham). *Fuel, Lond.*, 1957, **36** (3), 257-276.—After wet oxidation of the organic matter (cf. Ellington and Adams, *Brit. Abstr. B1*, 1952, 635),  $SiO_2$  is filtered off and determined gravimetrically, and Al, Fe, Ti, P and Mn are determined in separate aliquots of the filtrate by absorption spectrometry. Calcium, Mg, Na and K are isolated by pptn. in a further portion of the filtrate (after removal of Fe, Al, etc.), before being determined absorptometrically. Recognised procedures are used, with some modifications. Only 0.5 to 2.0 g of sample is required. Results are in fair agreement with those by methods in Fuel Res. Bd. *Survey Paper* 50, 1949, for Si, Fe and Mg; for Ca, Na and Ti they are slightly higher, and for K considerably higher. Flame-photometer results for Na and K are nearer the absorptometer results. Five complete analyses can be completed per man per week, against one by the F.R.B. methods.

A. R. PEARSON

See also Abstracts 1242, 1388, 1399, 1409, 1415.

### 3.—ORGANIC ANALYSIS

**1240. Spot-test analysis of organic substances.** F. Feigl, V. Gentil and C. Stark-Mayer (Lab. Prod. Mineral, Min. Agric., Rio de Janeiro, Brazil). *Mikrochim. Acta*, 1957, (3-4), 341-353 (in German).—Various methods have been developed. (i) *Anthraquinonesulphonic acids*—The dry sodium salt is heated under alkaline oxidising conditions to give a coloured polyhydroxyanthraquinone. (ii) *Secondary alcoholic groups*—The substance is heated with elementary sulphur to give the ketone and  $H_2S$ , which is detected by means of Pb acetate paper. (iii) *Succinic acid and succinimide*—Distillation of succinic acid or succinimide with zinc dust gives pyrrole, which forms a violet complex with *p*-dimethylaminobenzaldehyde. (iv) *1:2-Diketones*—By means of the benzilic acid re-arrangement, and subsequent evaporation to dryness in acid medium, disubstituted glycollic acids are formed. These are detected by formation of a coloured uranyl-acid-rhodamine B complex. (v)  *$\alpha$ -Alanine*—Decarboxylation and deamination with NaOBr gives acetaldehyde, which, even in the vapour form, gives a blue colour with sodium nitroprusside and piperidine. (vi) *Chloranil*—The ethereal or benzene solution and pyrrole give, after evaporation of the solvent, a blue coloration. Bromanil gives no coloration. (vii) *Naphthalene*—The vapour of naphthalene gives an intense brick-red colour at 50° to 60° with solid chloranil.

L. S. ADLER

**1241. New microchemical tests for carbon and nitrogen.** J. B. Niederl and J. A. Sozzi (New York Univ., N.Y., U.S.A.). *Mikrochim. Acta*, 1957, (3-4), 496-500 (in English).—The detection of C and N on the micro-scale is made more certain by a modification of the Lassaigne sodium fusion method. Both elements are detected as methylamine, which may be identified in several ways by standard tests. For the detection of C,  $(NH_4)_2SO_4$  is added to the original substance in the sodium fusion. For the detection of N, a C-donor, such as a N-free carbohydrate, is added. In each case the NaCN formed as a result of the fusion is acidified and the HCN so formed is reduced with Zn and HCl (10%).

L. S. ADLER



**1242. Rapid determination of the carbon content of organic and inorganic substances, by the use of conductivity measurements.** H. Malissa (Max Planck Inst. für Eisenforschung, Düsseldorf). *Mikrochim. Acta*, 1957, (3-4), 553-562 (in German).—The determination is based on the continuous measurement of the conductivity of a dilute soln. of NaOH through which CO<sub>2</sub>-containing gases are passing. With passage of gas, the conductivity decreases owing to the replacement of OH<sup>-</sup> by CO<sub>3</sub><sup>2-</sup> whose ionic mobility is considerably less than that of the OH<sup>-</sup>. The procedure is sensitive and rapid. The sample is burned and the gases are pumped through the conductivity cell at accurately adjusted constant flow-rates. Changes in conductivity are continuously recorded as amplified differential values against a blank cell through which no gases are passing. When the conductivity has decreased to a constant value, the combustion is complete. The carbon content of the sample is obtained by reference to a calibration curve. One determination takes about 2-5 min. (weighing time not included). Weights other than the sample weight are not required. L. S. ADLER

**1243. Rapid methods of micro-elementary analysis. XIV. Micro-determination of carbon and hydrogen in organofluorine compounds.** N. E. Gel'man, M. O. Korshun and N. S. Sheveleva (Inst. of Elementary Organic Compounds, USSR, Moscow). *Zhur. Anal. Khim.*, 1957, 12 (4), 526-533.—The method is based on rapid pyrolytic decomposition of the sample at 900° in a current of O. Absorption of F is obtained by use of MgO at 900°. Other halogens are absorbed by Ag at 550° to 600°. G. S. SMITH

**1244. Method for the simultaneous micro-analytical determination of the carbon, hydrogen and nitrogen content of organic compounds, by the use of a single sample.** W. Schöninger (Sandoz A.-G., Basle, Switzerland). *Mikrochim. Acta*, 1957, (3-4), 545-552 (in German).—The sample, mixed with CuO, is burned in a closed system, evacuated to less than 10<sup>-3</sup> mm (Hg). The resulting CO<sub>2</sub> and H<sub>2</sub>O are fractionally frozen out. The pressure of the N is then determined, and the N is pumped off. The CO<sub>2</sub> pressure is then determined and finally that of the water vapour. The empirical calibration of the apparatus enables the respective percentages or the atomic ratios of C, H and N to be calculated from these pressures. L. S. ADLER

**1245. Direct micro-determination of oxygen in organic compounds.** K. Bürger (Anal. Lab. Farbwerke Hoechst A.-G., Gendorf, Oberbayern). *Mikrochim. Acta*, 1957, (3-4), 313-317 (in German).—The direct determination of oxygen in organic compounds (Unterzaucher, *Ibid.*, 1956, 822) has been modified to increase its scope. Pure hydrogen replaces nitrogen as carrier gas. This has no effect on the quant. formation of CO when the thermal decomposition products are passed over C, and the iodimetric determination of the CO is not affected. The method may now be applied to organic compounds containing S, to organometallic compounds and to metal salts, provided that the metals are replaceable by hydrogen. Fluorine-containing compounds give inconsistent results. L. S. ADLER

**1246. Determination of non-aminoid nitrogen by the micro-Kjeldahl method. I. Aromatic nitro compounds.** T. S. Ma, R. E. Lang and J. D. McKinley, jun. (Microchem. Lab., New York Univ.,

N.Y., U.S.A.). *Mikrochim. Acta*, 1957, (3-4), 368-377 (in English).—The various modifications of the micro-Kjeldahl method for the determination of non-aminoid nitrogen have been assessed. The procedure described for aromatic nitro compounds consists in dissolving the sample in acetic acid and methanol, followed by reduction with zinc and HCl. Organic solvents are then removed by heating with conc. H<sub>2</sub>SO<sub>4</sub>. The product is incinerated with H<sub>2</sub>SO<sub>4</sub>, K<sub>2</sub>SO<sub>4</sub> and selenium metal catalyst, followed by standard micro-Kjeldahl distillation and titration. L. S. ADLER

**1247. Analysis for industry [organic fluorine compounds].** A. M. G. Macdonald. *Ind. Chem. Mfr.*, 1957, 33, 310-312.—Recent literature (1951 to 1957) on analytical techniques for the determination of F in org. compounds is reviewed. G. HELMS

**1248. A new lead chlorofluoride method for the determination of fluorine in organic compounds.** R. Belcher and A. M. G. Macdonald (Chem. Dept., Univ. Birmingham, England). *Mikrochim. Acta*, 1957, (3-4), 510-519 (in English).—A new semi-micro method is described. The compound is fused with alkali metal in a nickel bomb, and the fluoride is determined gravimetrically after pptn. as lead chlorofluoride, with a lead chloronitrate soln. as precipitant. The method can be applied to halogen- and N-containing compounds. Phosphate and sulphide interfere and must be separated before the pptn. A procedure in which ZnO is used is described. The whole procedure is suitable for routine work. L. S. ADLER

**1249. Solid-sample techniques in K-capture spectroscopy. Determination of chlorine and bromine.** W. Seaman, H. C. Lawrence and H. C. Craig (American Cyanamid Co., Bound Brook, N.J., U.S.A.). *Anal. Chem.*, 1957, 29 (11), 1631-1632.—A development of a previously reported method for use with liquid samples (*cf.* Hughes and Wilczewski, *Anal. Abstr.*, 1955, 2, 1533) is described. The solid sample is pressed into a thin disc, which is shaped to fit a holder adjacent to the X-ray source. Chlorine in chlorobenzanthrone and dichloroindanthrone may be determined in this way, with a <sup>56</sup>Fe source. Attempts to use this technique for bromine in bromobenzanthrone were unsuccessful, but a satisfactory modification consisted in suspending the powdered sample in heavy mineral oil (Nujol) and enclosing the suspension in a cell with beryllium windows. G. S. ROBERTS

**1250. A coulometric method for the determination of double bonds in small amounts of organic substances.** H. Flaschka and M. Hochenegger (Med. Chem. Inst., Univ. Graz, Austria). *Mikrochim. Acta*, 1957, (3-4), 587-593 (in German).—The method and apparatus described are based on the coulometric measurement of hydrogenation rates. The normal catalytic hydrogenation is carried out in a closed system in which a known pressure of H (slight excess over atmospheric) is maintained. As hydrogenation proceeds and the H pressure drops, it is made up by the introduction of electrolytic H until the original pressure is again obtained. Temperature, atmospheric pressure and current strength during electrolysis are kept constant. The duration of electrolysis is accurately timed with a stop-watch. By this means, on a sample in the range 5 to 20 mg, values for one double bond of 0.92 to 1.08 double bonds are obtained. A determination requires about 30 min. L. S. ADLER

**1251. Determination of  $\alpha$ -epoxides containing a tertiary carbon atom via catalytic isomerisation with zinc bromide.** A. J. Durbetaki (Food Machinery and Chem. Corp., Buffalo, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1666-1668.—The  $\alpha$ -epoxide is converted into its corresponding aldehyde by catalytic isomerisation with  $\text{ZnBr}_2$  in benzene; the resulting aldehyde is determined gravimetrically as the 2:4-dinitrophenylhydrazone. This procedure is used as the basis for the determination of  $\alpha$ -pinene oxide, camphene oxide,  $\alpha$ -methylstyrene oxide and 1:2-diisobutylene oxide. The recoveries of these compounds were greater than 98%. G. P. COOK

**1252. Densities and refractive indices for glycol-water solutions. Triethylene glycol, dipropylene glycol and hexylene glycol [2-methylpentane-2:4-diol].** T.-T. Chiao and A. R. Thompson (Univ. R.I., Kingston, U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1678-1681.—Densities at 25° and refractive indices at 20° and 25° were determined for mixtures of triethylene glycol, dipropylene glycol or hexylene glycol with water. The refractive index determinations give results for composition accurate to within  $\pm 0.1\%$  by wt. for all 3 glycols except for hexylene glycol at a glycol content  $>95\%$  when the accuracy is within  $\pm 0.4\%$ . Owing to points of inflection in two of the density curves, the usefulness of the density data varies with composition. The applicability of the Eykman equation was tested at 20° and 25° for the pure compounds and was found to be satisfactory. G. P. COOK

**1253. Paper chromatography of homologous saccharides. Selection of solvent components and solvent proportions.** J. A. Thoma and D. French (Iowa State College, Ames, U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1645-1648.—A procedure is described which allows the selection of solvent proportions of ternary systems producing the highest mobilities of homologous saccharides concomitant with their complete separation on chromatograms. The solvent components of the ternary systems are selected for desirable physical properties. G. P. COOK

**1254. Chromatographic analysis of mixtures of formaldehyde, acetaldehyde and acrolein.** L. Nebbia and F. Guerrieri ("G. Donegani" Res. Inst., Montecatini, Novara, Italy). *Chim. e Ind.*, 1957, **39** (9), 749-750.—The aldehydes are converted into their 2:4-dinitrophenylhydrazones (**I**) and then separated in  $\text{CHCl}_3$  or toluene soln. by adsorption on partially hydrated  $\text{MgO}$ . The coloured **I** are then developed by eluting with pyridine. The colours of the adsorbed **I** of acetaldehyde, propionaldehyde, benzaldehyde, salicylaldehyde, cinnamaldehyde, furfuraldehyde, acetone, ethyl methyl ketone and cyclohexanone are given. A. G. COOPER

**1255. Determination of higher aliphatic aldehydes in presence of ketones and fatty acids.** L. D. Metcalfe and A. A. Schmitz (Armour and Co., Chicago, Ill., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1676-1678.—The method is based on the ease with which aliphatic aldehydes are oxidised to fatty acids by a mixture of 3%  $\text{H}_2\text{O}_2$  and standard  $\text{NaOH}$ . The amount of standard alkali consumed in the reaction is a measure of the aldehyde content. Free fatty acids in the sample are titrated separately and a correction for these is made. The accuracy is generally well within  $\pm 5.0\%$  even in the presence of considerable quantities of ketone, and good agreement with the results by another method were obtained. G. P. COOK

**1256. Determination of sorbic acid and its salts.** W. Diemair, K. Franzen and A. Sieglitz (Univ.-Inst. für Lebensmittelchemie, Frankfurt a.M., Germany). *Naturwissenschaften*, 1957, **44** (6), 180-181.—In the presence of biological material the following colorimetric method has yielded satisfactory results. The aq. soln. containing sorbic acid is extracted repeatedly with diethyl ether and the combined extracts are evaporated to dryness. The residue is dissolved in 5 ml of acetic anhydride, and to 1 ml of this soln. are added 30 mg of 3-ethyl-2-methylthiobenzothiazolium toluene-*p*-sulphonate and 50 mg of anhydrous  $\text{Na}$  acetate, and the mixture is heated on the oil bath with constant stirring at 135° for 15 min. Methanol (10 ml) is then added and the soln. is allowed to cool. The extinction is then measured in a spectrophotometer at 652  $\mu$  in a 0.25-cm cell. A calibration curve over the working range can be constructed in a similar manner. E. KAWERAU

**1257. Paper-chromatographic determination of hydroxystearic acids.** K. Winsauer (Univ. Graz, Austria). *Mikrochim. Acta*, 1957, (3-4), 480-484 (in German).—The detection of hydroxystearic acids and the separation of the di-, tetra- and hexa-hydroxy acids are described. Detection is based on the reaction with  $\text{KIO}_4$ . The acids are oxidised by the periodate so that this, being itself reduced, is no longer available for the liberation of iodine from  $\text{KI}$  soln. When the chromatogram is developed with  $\text{KI}$  and starch, the hydroxy acids appear as white spots on a blue background. Separation is effected by using a mobile phase consisting of a mixture of butanol, ethanol, aq.  $\text{NH}_3$  and water. Mono- and tri-hydroxystearic acids cannot be separated by this means in that they are, respectively, indistinguishable from the di- and tetra-hydroxy acids. A method is given for the detection of saturated fatty acids in the presence of oleic, linoleic and linolenic acids by converting the unsaturated acids into their corresponding hydroxystearic acids. L. S. ADLER

**1258. Micro-detection and colorimetric micro-determination of oxalate ions by an activated reaction.** G. Almássy and I. Dezső (Inst. Med. Chem., Debrecen, Hungary). *Acta Chim. Acad. Sci. Hung.*, 1957, **11** (1-2), 7-13 (in English).—Oxalate ions can be detected by their activating effect on the reaction between  $\text{VV}$  and aniline in acid soln. In the presence of oxalate ions a greenish-blue colour appears within a few minutes. The limit of detection is 1  $\mu$ g of oxalate ions in 1 ml of soln. Amounts of up to 1 mg of  $\text{Co}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Cr}^{3+}$  and  $\text{Cu}^{2+}$ , or large quantities of other cations, do not interfere. The presence of more than 5 mg of  $\text{PO}_4^{3-}$  or  $\text{F}^-$  reduces the sensitivity to 50  $\mu$ g of oxalate ions in 1 ml of soln.; other anions do not interfere. Oxalate ions can also be determined by means of the reaction between  $\text{Cr}^{\text{VI}}$  and  $\text{Mn}^{\text{II}}$ , which continues as long as oxalate ions are present. The residual  $\text{Cr}^{\text{VI}}$  is determined photometrically with diphenylcarbazide; by this method, oxalate can be determined in the concn. range 0.5  $\mu$ g to 10  $\mu$ g per ml. The max. permissible amounts of foreign ions are reported; none interfere in amounts of  $<1$  mg. The errors reported lie between  $\pm 9\%$ . G. BURGER

**1259. Determination of acetic anhydride.** E. N. Novikova and L. N. Petrova (All-Union Sci. Res. Inst. of Synthetic and Natural Perfumes, Moscow). *Zhur. Anal. Khim.*, 1957, **12** (4), 534-539.—The

hydrolysis of acetic anhydride is catalysed both by pyridine and by  $\text{H}_2\text{SO}_4$ . Two methods of determining acetic anhydride are given, that based on the use of  $\text{H}_2\text{SO}_4$  is as follows. The sample ( $\approx 1$  to 1.5 g of anhydride) is mixed with 8 to 10 ml of acetic acid containing 0.2 to 0.3% of  $\text{H}_2\text{SO}_4$  and then with water sufficient to give an excess of 20 to 25% over that required to combine with the anhydride. After 5 min. at room temp. in a closed flask the soln. is titrated with the Karl Fischer reagent. A blank is carried out at the same time. To determine the anhydride in glacial acetic acid, it is sufficient to determine the moisture content before, and after addition of 0.2 to 0.3% of  $\text{H}_2\text{SO}_4$ .

G. S. SMITH

**1260. Titration of nitro-aromatic amines as acids.** J. S. Fritz, A. J. Moyer and M. J. Richard (Iowa State College, Ames, U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1685-1688.—Anilines substituted in the 2- and 4- or 2-, 4- and 6-positions with at least two nitro groups or with one nitro group and one or more chlorine atoms, can be titrated as acids in pyridine with *n*-butyltriethylammonium hydroxide as the titrant. Diphenylamine derivatives with at least one nitro group in the 4-position can be titrated in a similar manner. Trinitrotoluene and trinitrobenzene can also be titrated as monobasic acids. In several cases mixtures can be resolved. Recoveries were generally > 99%.

G. P. COOK

**1261. Long-chain amines. Versatile acid extractants.** F. L. Moore (Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1660-1662.—Soln. of long-chain amines are shown to be satisfactory extractants for mineral, many organic and complex metal acids. Two of the most promising amines studied were tribenzylamine (I) and methyldi-*n*-octylamine (II). Recoveries of many acids were satisfactory with II, and anionic Po and  $\text{PuO}_2^{2+}$  were extracted with I. Extraction of anionic Zr and Pa with II was  $\approx 99\%$  under optimum acidity conditions; Hf was also extracted under similar conditions. A strong analogy exists between anion-exchange resins and soln. of long-chain amines. Some of the advantages of the liquid-liquid extraction technique are discussed.

G. P. COOK

**1262. Determination of carboxylic acid amides by saponification.** B. V. Ioffe and Z. I. Sergeeva (Chem. Inst., Leningrad State Univ.). *Zhur. Anal. Khim.*, 1957, **12** (4), 540-544.—With easily saponifiable amides, such as formamide, methylformamide, dimethylformamide, etc., the sample containing the amide (5 mM) is boiled for 1.5 to 2 hr. with 20 ml of 0.5 *N* ethanolic KOH and then titrated with 0.5 *N* ethanolic acetic acid, with thymolphthalein as indicator. The amine formed need not be removed since it does not affect the indicator. With difficultly saponifiable amides, such as diethylformamide, acetamide, ethylacetamide, etc., the sample (8 to 10 mM) is boiled for 3 to 4 hr. with 20 ml of *N* KOH in ethanol, and titrated potentiometrically, with a glass electrode, if the soln. is too dark in colour for titration with an indicator.

G. S. SMITH

**1263. Cryoscopic determination of material not reacting with sulphuric acid in aromatic hydrocarbons.** M. D. Tilicheev and E. I. Goisa (All-Union Sci. Res. Inst. for the Transport, Storage and Use of Petroleum Products, Moscow). *Zhur. Anal. Khim.*, 1957, **12** (4), 550-555.—The sample is mixed

with cyclohexane of known crystallisation point so that the mol. proportion of aromatic hydrocarbons is  $\approx 50\%$ . The mixture is treated with conc.  $\text{H}_2\text{SO}_4$  to remove aromatic hydrocarbons completely, leaving the unattacked impurities in the cyclohexane. Their concn. is found by determining the crystallisation point of the solution.

G. S. SMITH

**1264. Determination of the *p*-xylene content in the xylene fraction by a cryoscopic method.** M. Popowicz (Dept. of Contact Synthesis, Inst. of Gen. Chem., Warsaw). *Chem. Anal., Warsaw*, 1957, **2** (4), 358-365.—The amount of *p*-xylene in the unknown mixture is read from a standard f. p. curve obtained with known mixtures of pure *p*- and *m*-xylene. It is shown that amounts of *o*-xylene, ethylbenzene, toluene and mesitylene such as are likely to occur in a *m*-xylene isomerisation mixture do not interfere.

T. M. M.

**1265. Micro-determination of organic compounds, using a new type of relaxational coulometer. II. Micro-determination of phenol and cresols.** K. Sykut (Katedry Chem. Nieorgan. Wydziału Mat.-Fiz.-Chem. UMCS, Lublin). *Ann. Univ. M. Curie-Skłodowska, AA*, 1955, **10** (3), 25-34.—The coulometer described in Part I (cf. *Anal. Abstr.*, 1957, **4**, 2056) was slightly modified. The mean error of 50 given determinations was  $\approx 0.5 \mu\text{g}$ , the amounts determined varying between 2 and 100  $\mu\text{g}$  (in dilutions of 1 in  $10^3$  to 1 in  $10^2$ ). The method is based on bromination of the sample with excess of Br, followed by treatment with excess of KSCN, and coulometric determination of the unchanged KSCN. The method can be used for other org. compounds which cannot be determined by direct bromination, and it is suggested that the sensitivity may be increased for the determination of amounts < 1  $\mu\text{g}$ .

J. M. L. MESSING

**1266. Detection of eugenol, isoeugenol and vanillin.** F. Meyer and E. Meyer (Pharmacology Inst., Hamburg Univ., Germany). *Arch. Pharm., Berlin*, 1957, **290** (3), 109-117.—The detection of small amounts is possible with the following methods. (i) Adsorption on Frankonit KL columns on which eugenol gives a blue-green and isoeugenol a carmine-red coloration. As little as 0.50 mg of eugenol and 0.1 mg of isoeugenol can be detected when both substances are present. The colour reaction is especially sensitive in acid medium. Vanillin does not interfere. (ii) Paper chromatography, with either *n*-propanol or *n*-butanol with 10% of water as solvent, S. & S. paper No. 2045b and 2-naphthylamine acetate as spray, is a more sensitive method. It is possible to detect 0.05 mg of eugenol (orange), 0.01 mg of isoeugenol (yellow) and 0.005 mg of vanillin (yellow). (iii) Each of the three compounds has a characteristic u.v. absorption and, by spectrophotometric examination, purity can be tested and concn. of 0.0005 mg per ml be quant. determined.

J. WATT

**1267. Oxidative metabolism of phenylacetic acid by *Penicillium chrysogenum* Q 176. XI. Colorimetric micro-determination of phenylacetic acid.** Masao Isono and Kikuko Yoshino (Osaka Factory, Takeda Pharm. Ind. Ltd., Jusonishino-cho, Higashiyodogawa-ku, Osaka). *J. Agric. Chem. Soc. Japan*, 1956, **30** (12), 806-809.—Phenylacetic acid (I) is quant. nitrated with 0.2 ml of 0.2%  $\text{KNO}_3$  in conc.  $\text{H}_2\text{SO}_4$ ; the excess of  $\text{KNO}_3$  does not interfere with the succeeding reduction and diazotisation. The diazonium salt is treated with

3-diethylaminoethyl-1-naphthylamine oxalate (**II**), and is determined photometrically at 560 m $\mu$ . *Procedure*—The sample (1 ml) containing 50 to 500  $\mu$ g of **I** is mixed with 10 N H<sub>2</sub>SO<sub>4</sub> (0.1 ml) and Na<sub>2</sub>SO<sub>4</sub> (0.3 g) and extracted with toluene (5 ml). A 1-ml portion of the organic layer is evaporated to dryness, and nitrated at 42° for 15 min. The product is dissolved in water (1 ml), treated with 6 N HCl (1.0 ml) and Zn (0.2 g), and diluted with 4 ml of water. A 1-ml portion is treated successively with 0.05% NaNO<sub>2</sub> (0.5 ml) and ammonium sulphamate (0.1%, 0.5 ml), and the product is photometrically determined after addition of 0.1% **II** soln. (0.5 ml) and ethanol (2.5 ml). No interference results from penicillin G, cornsteep liquor or *o*-hydroxyphenylacetic acid. K. SAITO

1268. **Bromimetric determination of cinnamic acid and its derivatives.** M. R. Verma, K. C. Agrawal and S. D. Paul (Nat. Phys. Lab., New Delhi). *J. Sci. Ind. Res., B, India*, 1957, **16** (5), 213-215.—A study has been made of the number of atoms of Br absorbed by cinnamic acid and its derivatives from a bromide-bromate mixture, with a view to developing a bromimetric method for the quantitative determination. The effect of temp., pH and excess of reagent has been examined, and it has been found that cinnamic acid and *m*-coumaric acid absorb an integral number of atoms of Br. A bromimetric method for the determination of these two substances is therefore feasible. C. A. SLATER

1269. **Infra-red photometric analysis of toluic acids, benzenedicarboxylic acids and xylyl chlorides.** Akito Kobayashi, Shizuo Nagahama and Saburo Akiyoshi (Dept. of Applied Chem., Faculty of Engng, Kyushu Univ., Hakozaki, Fukuoka). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1956, **59** (8), 974-976.—Infra-red absorption spectra of individual isomers of ethyl toluate (**I**), dimethyl and diethyl benzenedicarboxylates (**II**) and of xylyl chloride (**III**) were measured in the range 9 to 15  $\mu$ . The analysis of isomers of **I** can be satisfactorily made by the use of the bands at 13.28, 13.44 and 13.59  $\mu$  (for *p*-, *m*- and *o*-isomers, respectively) as key bands. The mixture (1 to 2 g) of the isomeric toluic acids is esterified by refluxing with ethanol (10 to 20 ml) and conc. H<sub>2</sub>SO<sub>4</sub> (0.2 to 0.4 ml). No key bands were found that would enable simultaneous determination of the three isomers of **II** to be made. The diethyl esters of the *m*- and the *p*-isomers of **II** are analysed with the bands at 7.67, 8.01 (both for *m*-) and 7.87 (*p*-)  $\mu$ . The isomers of **III** are similarly analysed with three key bands at 13.03, 12.70 and 12.25  $\mu$  (for *o*-, *m*- and *p*-isomers, respectively). K. SAITO

1270. **Paper chromatography of nitrotoluidines. II. Separation and identification of isomeric mononitro derivatives of *p*-toluidine.** A. Waksmundzki, J. Ościk and Z. Frelek (Katedry Chem. Fiz. Wydziału Mat.-Fiz.-Chem. UMCS, Lublin). *Ann. Univ. M. Curie-Skłodowska, A*, 1955, **10** (2), 17-24.—Two mononitro derivatives of *p*-toluidine were separated by the use of Whatman No. 3 paper with humidity coefficient 1.48 to 1.51, and water-saturated *n*-hexane for developing the chromatogram. The *R<sub>F</sub>* value of 2-nitro-*p*-toluidine was 0.50 and of 3-nitro-*p*-toluidine 0.78. By using the same method to separate six mononitro derivatives of *o*- and *p*-toluidine [3-nitro-*o*-toluidine, 4-nitro-*o*-toluidine (**I**), 5-nitro-*o*-toluidine, 6-nitro-*o*-toluidine, 2-nitro-*p*-toluidine (**II**), and 3-nitro-*p*-toluidine] it was found that **I** (*R<sub>F</sub>* 0.46) and **II** (*R<sub>F</sub>* 0.50) were not

separated. A successful separation was achieved by drying the chromatogram in air, re-moistening it with 5% aq. formic acid soln., drying to an humidity coefficient of 1.50, and developing the chromatogram at right angles to the first one, again with *n*-hexane. The *R<sub>F</sub>* values were then 0.27 and 0.10 for **I** and **II**, respectively. J. H. L. MESSING

1271. **Determination of microgram amounts of aniline, methylaniline and dimethylaniline in admixture.** A. A. Belyakov and N. V. Gorbyleva (Gorki Inst. of Work Hygiene and Occupational Diseases). *Zhur. Anal. Khim.*, 1957, **12** (4), 545-549.—Separate determinations of aniline, dimethylaniline and the sum of methylaniline and aniline are made. For aniline, the sample soln. (2 ml) is treated with 0.1 ml of 40% acetic acid soln. and 0.3 ml of a reagent containing 20 mg of NaNO<sub>2</sub> and 35 mg of NaBr. After 5 min., 0.2 ml of 10% aq. NH<sub>3</sub> is added then, > 1 min. after, 0.25 mg of 1-naphthol dissolved in 0.5 ml of ethanol, followed by 0.1 ml of 40% NaOH and dilution to 5 ml with ethanol. After 10 min. the extinction at 533 m $\mu$  is measured. With 1 to 40  $\mu$ g of aniline in 2 ml the error is  $\pm \approx 6\%$ . For dimethylaniline, 4 ml of the soln. acidified with 0.1 ml of 15% acetic acid is mixed with 25 mg (two drops) of acetic anhydride, 1 ml of 10% Na acetate soln. and 0.35 ml of a reagent soln. prepared by mixing a soln. of 25 mg of *p*-nitroaniline in 20 ml of 20% acetic acid soln. with 4 ml of 1% NaNO<sub>2</sub> soln. The soln. is diluted to 10 ml with ethanol and its extinction at 465 m $\mu$  is measured after 10 min. With 1  $\mu$ g of dimethylaniline in 4 ml the error is  $\pm \approx 6\%$ . For methylaniline and aniline together, 2 ml of the soln. is treated with 0.9 ml of 15% aq. ethanol and 0.5 ml of a 6% soln. of chloramine B. After 5 min., 0.1 ml of 0.1 N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, 0.1 ml of 6% NaOH soln. and 0.4 ml of water containing 24 mg of phenol are added. After 20 min. the extinction is measured at 619 m $\mu$ . With 1  $\mu$ g of methylaniline or aniline in 2 ml the error is  $\pm \approx 20\%$ . The content of methylaniline is found by difference. To determine the bases in air, the sample is passed at a rate of 1 litre per minute through three absorption vessels containing 5 ml of 1% ethanol, the third containing also 0.3 ml of 40% acetic acid. Aniline and methylaniline are retained in the first two vessels, and dimethylaniline in all the vessels. G. S. SMITH

1272. **A colour reaction of euchroic acid serving to identify mellitic acid.** M. Chaigneau (Lab. de Prof. Lebeau, Fac. de Pharm., Paris). *Ann. Pharm. Franc.*, 1957, **15** (4), 224-226.—Wöhler's reaction in which ammonium euchroate (diammonium 1:2:4:5-bisimidodicarbonylbenzene-3:6-dicarboxylate) (formed from ammonium mellitate by heating at 150°) or its free acid gives with zinc a blue colour turning purple with alkali, is also given when benzene-1:2:4:5-tetracarboxylic acid (pyromellitic acid) yields 1:2:4:5-bisimidodicarbonylbenzene under similar conditions. E. J. H. BIRCH

1273. **Paper chromatography of isothiocyanates.** S. Fişel, F. Modreanu and A. Carpov. *Stud. Cercet. Stiint. Chim., Iasi*, 1956, **7** (2), 19-23.—The allyl, phenyl, *o*-, *m*- and *p*-tolyl, and 1- and 2-naphthyl isothiocyanates are separated and identified by the coloured thiosemicarbazides formed by their reacting with 2:4-dinitrophenylhydrazine (**I**). An ethanolic soln. of an isothiocyanate (0.5%) (1 ml) is added to an ethanolic soln. of **I** (0.05%) (2 ml) and heated under reflux for 10 to 15 min. The spots



are best developed with a mixture of ethanol, pyridine and water (4:0.5:5.5, by vol.).  $R_F$  values are given for the above thiosemicarbazides, using various paper techniques. H. SHER

**1274. The determination and characterisation of biologically active pyrroles.** G. Müller (Med. Klin., Charité Klin.-chem. Labor., Berlin). *J. prakt. Chem.*, 1957, **4**, [Series 4], 179-189.—Pyrroles and indoles in acetic acid were condensed with isatin by boiling for 5 min. with 50%  $H_2SO_4$ ; the dye formed was pptd. by the addition of 2 N NaOH. Characteristics of compounds formed by a number of pyrrole and indole derivatives are reported. N. E.

**1275. Spectrophotometric determination of small quantities of some individual pyridine bases by successive extractions.** T.-C. L. Chang and C. Karr, jun. (Bureau of Mines, Morgantown, W. Va., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1617-1619.—The method is based on the extraction of pyridine bases from aq. soln. with isooctane and subsequent spectrophotometric determination of the bases. Normally, several extractions are required for complete recovery, but this is overcome by applying the distribution coeff. calculated from two extractions. From these, the total concn. of the bases in the original sample are calculated, and recoveries of 98 to 102% were obtained from single and mixed pyridine base standards. The limiting concn. for this accuracy is about 0.01 g per litre. G. P. COOK

**1276. Some new microchemical reactions of piperazine and of 2:5-dimethylpiperazine and their application to micro-analysis.** B. Berisso (Inst. Invest. Microquim., Univ. Litoral). *Mikrochim. Acta*, 1957, (3-4), 296-305 (in Spanish).—The study of the properties of bismuth potassium iodide led to new micro-analytical pptn. reactions suitable for the characterisation of piperazine and 2:5-dimethylpiperazine and for their differentiation. The proposed procedure permits the detection of both materials in admixtures of all proportions. The compounds formed differ in colour, crystal form and detection sensitivity. Other microchemical reactions, also suitable for the characterisation of these compounds, were studied. L. S. ADLER

**1277. Non-aqueous titration of 1:4-disubstituted piperazines.** L. L. Ciacchio, S. R. Missan, W. H. McMullen and T. C. Grenfell (Chas. Pfizer & Co. Inc., Brooklyn, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1670-1673.—Solvents tested were glacial acetic acid, methyl cyanide and nitromethane, with  $HClO_4$  in acetic acid as the titrant. Two potentiometric end-points were found with methyl cyanide and nitromethane, and one end-point with glacial acetic acid. G. P. COOK

**1278. Quantitative estimation of 3-amino-1:2:4-triazole.** F. O. Green and R. N. Feinstein (Argonne Nat. Lab., Lemont, Ill., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1658-1660.—3-Amino-1:2:4-triazole in trichloroacetic acid soln. is diazotised and the diazo compound formed is coupled with chromotropic acid to form a coloured derivative, which is measured absorptiometrically at 525 m $\mu$ . The relationship between absorption and concn. is not linear, but follows a smooth curve. The sensitivity is  $\approx 10 \mu g$  and the average deviation is  $\approx 2\%$  over the concn. range of 10 to 125  $\mu g$ . Use of these

reagents as sprays enables as little as 0.2  $\mu g$  of aminotriazole to be detected on paper chromatograms. G. P. COOK

**1279. Spot tests based on Nencki synthesis of rhodanine (rhodanic acid).** F. Feigl and V. Gentil (Lab. da Prod. Mineral, Min. da Agric., Rio de Janeiro, Brazil). *Anal. Chem.*, 1957, **29** (11), 1715-1716.—If rhodanine (I) is treated with 1:2-naphthaquinone-4-sulphonic acid in alkaline soln., a water-sol. blue para-quinoidal compound is formed. This reaction allows I to be detected in a spot test with an identification limit of 0.6  $\mu g$ . The reaction may also be used to detect the reactants of the Nencki synthesis of I, i.e., chloroacetic acid and  $NH_4SCN$ . Since other similar substances can be used to prepare I by this method, the reaction can be extended to include them, viz. alkali thiocyanates, thiourea and its N-mono-alkylated and -arylated derivatives, bromoacetic acid (and probably iodoacetic acid); cyanamide and its salts can be detected through their ready conversion into thiourea. G. S. ROBERTS

**1280. Determination of closed flash-point of petroleum products by means of the Pensky-Martens apparatus.** British Standards Institution (2 Park St., London). B.S. 2839:1957, 11 pp.—The method is intended for petroleum products having a flash-point above 120° F. It should not be used for cut-back bitumens. O. M. WHITTON

**1281. Method for determination of tetraethyl-lead in gasoline (hydrochloric acid method).** British Standards Institution (2 Park St., London). B.S. 2878:1957, 9 pp.—The tetraethyl-lead is converted into lead chloride and extracted from the sample by refluxing with conc. HCl. The acid extract is evaporated to dryness, any organic material present is removed by oxidation with  $HNO_3$ , and the Pb is determined gravimetrically as  $PbCrO_4$ . Results of duplicate tests should not differ by more than the following amounts: repeatability 0.03 + 0.026A, and reproducibility 0.06 + 0.032A, where A = mean tetraethyl-lead content in ml per imp. gallon. O. M. WHITTON

**1282. Determination of acid and base numbers (neutralisation value) of petroleum products by colour indicator titration.** British Standards Institution (2 Park St., London). B.S. 2834:1957, 11 pp.—The method is applicable to acids or bases with dissociation constants in water  $> 10^{-9}$ ; extremely weak acids or bases with dissociation constants  $< 10^{-9}$  do not interfere, but salts react if their hydrolysis constants are  $> 10^{-9}$ . To determine the total acid or strong base number, the sample is dissolved in a mixture of benzene and isopropyl alcohol containing a little water, and the resulting single-phase soln. is titrated at room temp. with standard alcoholic base or alcoholic acid soln., respectively, to the end-point, with 1-naphtholbenzein soln. as indicator. To determine the strong acid number, a separate portion of the sample is extracted with hot water and the aqueous extract is titrated with KOH soln., with methyl orange as indicator. O. M. WHITTON

**1283. Analysis of hydrocarbon mixtures. I. Partition of diesel oil fractions and ascertainment of constituents.** G. Kühnhanss, H. Rösner, E. Hüttig, M. Wagner and G. Tischendorf (VEB Hydrierwerk, Zeitz, Germany). *Erdöl u. Kohle*, 1957, **10** (6), 372-375.—In a systematic examination of hydrogenation products of low-temp. tar from brown coal,

the normal paraffins were isolated as urea adducts by treatment with a 4-fold excess of urea in methanol; aromatics were separated from alkylnaphthenes by extracting the residue, diluted with hexane (1 vol.), with  $\text{SO}_2$  at  $-35^\circ$ . Each group was then distilled at 3 mm (Hg) through a packed column (6 theoretical plates) to yield five or six fractions. The paraffin fractions were each crystallised in six fractions from dichloroethane at  $5^\circ$  intervals between  $-25^\circ$  and  $10^\circ$ . The alkylnaphthene raffinate fractions were each fractionated by chromatographic adsorption and elution with light petroleum and acetone at 2.1 to 2.4 atm. pressure, yielding 176 fractions. The aromatic extract fractions were each fractionated by thermal diffusion at constant temp. gradient in an annulus 0.2 mm wide between two coaxial brass tubes. The paraffin fractions were classified according to their structure (*cf.* Gross and Grodte, *Oel u. Kohle*, 1942, **38**, 419). The compositions of the paraffin and naphthene fractions were calculated by the  $n-d-M$  method, and those of the aromatic fractions by the method of Martin and Sankin (*Brit. Abstr. C*, 1953, 490). A diesel oil fraction yielded paraffins ( $\approx 11\%$ ) containing 13 to 23 carbon atoms, alkylnaphthenes ( $\approx 59\%$ ) containing 1 to 4 fused naphthenic rings and side-chains of 4 to 23 carbon atoms, and aromatics containing 1 or 2 naphthenic rings fused with 1 or 2 aromatic rings with side-chains of 3 to 6 carbon atoms.

A. R. PEARSON

**1284. Separation of organic sulphur compounds by liquid thermal diffusion.** C. J. Thompson, H. J. Coleman, C. C. Ward and H. T. Rall (Bureau of Mines, Bartlesville, Okla., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1601-1611.—Liquid thermal diffusion was applied to the separation and identification of sulphur compounds in crude oils and the procedure was used for some 50 binary mixtures of pure compounds as well as for S-containing petroleum distillates. Continuous withdrawal of the sample from the extreme ends of the column was superior to batch operation in that a greater percentage of a given charge could be separated. Separation by thermal diffusion can often be achieved on materials with identical boiling-point and mol. wt., without the addition of solvents, and at the same time avoiding exposure to severe thermal conditions. An apparatus and technique for charging the column, permitting air-free operation, and a semi-micro column of capacity 0.95 ml are described.

G. P. COOK

**1285. Investigations of tritium "Bremsstrahlung" as a means of determining sulphur and tetraethyl-lead in hydrocarbons.** M. M. Kannuna (A.E.R.E., Harwell, England). *J. Inst. Petrol.*, 1957, **43**, 198-202.—A source of X-rays of wavelength 0.68 Å upwards was prepared by adsorption of tritium on a vacuum-evaporated film of Zr; it had an activity of 114 millicuries over an area of 1.3 sq. cm. This radiation was passed through a brass cell containing a layer 5.09 mm thick of the hydrocarbon liquid to be investigated, and the emergent radiation was collected by a Mullard MX.118 Geiger counter with auxiliary counting equipment. The counting rate was corrected for background and for the artificial dead time of 400 micro-sec. introduced by the quench unit. From measurements of the absorption of the "Bremsstrahlung" in pure benzene, toluene, *o*-xylene, cyclohexane and *n*-heptane, the mass absorption coeff. for H and C were calculated to be  $0.9 \pm 0.04$  sq. cm per g and  $4.21 \pm 0.2$  sq. cm per g, respectively. Thiophen

[0.5 to 5% (w/w)] added to benzene was determined with an accuracy of 0.033% (w/w) for a standard deviation of  $\pm 0.3\%$  in the counting rate. For tetraethyl-lead in *n*-heptane [0.1 to 1% (v/v)], the corresponding figure was 0.004% (v/v).

A. JOBLING

**1286. Determination of drop-point of petroleum grease.** British Standards Institution (2 Park St., London). B.S. 2877:1957, 8 pp.—The sample, contained in a metal cup, is heated until a drop falls from the orifice of the cup. The results of duplicate tests should not differ by more than the following amounts—repeatability  $6^\circ\text{F}$ , and reproducibility  $12^\circ\text{F}$ .

O. M. WHITTON

**1287. Reduction of coal samples taken for determination of total moisture content.** A. Crawford and D. H. Ward (Nat. Coal Bd., N.W. Division). *J. Inst. Fuel*, 1957, **30**, 314-321.—The moisture sample (*cf.* B.S. 1017:1942) should be air-dried before it is crushed from  $\geq 13$  mm to  $\geq 2.8$  mm for the lab. sample, otherwise moisture may be lost in the crushing. Air-drying is unnecessary when moisture is determined by distillation from 1 kg of the  $\geq 13$ -mm sample. If the preliminary size reduction is limited to  $\geq 19$  mm instead of  $\geq 13$  mm, and increments of double weight are taken, the air-drying of the 2-kg sample is less complete, but the total moisture is unaffected. Crushing may be done by jaw or rotary crusher provided the latter is not ventilated. A "low-rank" coal may require air-drying before crushing to  $\geq 13$  mm.

A. R. PEARSON

**1288. Moisture test of coal in relation to sampling and analysis.** M. Hissink and D. J. W. Kreulen (Kreulen Lab., Rotterdam). *BrennstChemie*, 1957, **33** (13-14), 217-218.—The gross sample is reduced directly to a laboratory sample of  $\approx 5$  kg of  $\geq 4$  mm particle size by crushing and quartering. By working quickly, with an unventilated mill, and avoiding heating the sample, loss of moisture during the reduction is negligible. Two 300-g portions are dried at  $105^\circ$  to  $110^\circ$  for 2 to 3 hr. for moisture determination; the rest of the sample is also dried and reduced to  $< 0.2$  mm for determination of ash, S, volatile matter, etc., a simultaneous moisture test being carried out to check any possible atmospheric effects. The method is quicker than the two-stage process involving air-drying of the gross sample, and on moisture contents of 3 to 12% differences of  $\geq 0.3\%$  are found.

A. R. PEARSON

**1289. Methods for the analysis and testing of coal and coke.** 2. Total moisture of coke. British Standards Institution (2 Park St., London). B.S. 1016: Part 2: 1957, 8 pp.—In this revised Standard, the indirect method for the determination of moisture in the coke "as sampled" is described.

**3. Proximate analysis of coal.** British Standards Institution. B.S. 1016: Part 3: 1957, 25 pp.—Methods are described for the determination of moisture, volatile matter and ash in the analysis sample ground to pass a 72-mesh B.S. test sieve. Tolerances are indicated.

**4. Proximate analysis of coke.** British Standards Institution. B.S. 1016: Part 4: 1957, 14 pp.—Methods are described for the determination of moisture, volatile matter and ash in the analysis sample ground to pass a 72-mesh B.S. test sieve. Tolerances are indicated.

**5. Gross calorific value of coal and coke.** British Standards Institution. B.S. 1016: Part 5: 1957, 28 pp.—Revised standard procedures are given for

the determination of the calorific value of coal or coke at 25° by burning a sample (1 g, 72-mesh) in O in a bomb calorimeter of known heat capacity. The method of determining, periodically, the effective heat capacity (water equivalent) of the system is specified more closely (a Ni-Cr foil crucible is recommended for ensuring complete combustion of benzoic acid). The cooling correction should be calculated by either the Regnault-Pfaundler or the Dickinson formula; an example of each calculation is given. The initial pressure of O in the bomb (250 ml to 350 ml) should be 25 atm. for coal and 30 atm. for anthracite, coke or hard-to-burn coals. To obviate failure through inter-crystalline corrosion, specified materials only are to be used in the manufacture of the components of the bomb. Highly accurate fixed-scale thermometers are specified (the Beckmann type is excluded) and crucibles may be of platinum, nickel-chromium or silica (a different shape is used for coke samples).

O. M. WHITTON  
W. J. BAKER

**1290. Determination of sulphur in coal and coke.** R. A. Mott and H. C. Wilkinson (Brit. Coke Res. Ass., London). *Brennst. Chem.*, 1957, **38** (13-14), 212-215.—The Eschka method and the Sheffield high-temp. combustion method give consistent results for S if the experimental details given are observed. Conductimetric titration and other modifications of the latter method are not advantageous and require more time.

A. R. PEARSON

**1291. Quinone character of oxidation products of a bituminous coal.** C. R. Kinney and D. L. Love (Pennsylvania State Univ., University Park, U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1641-1645.—Humic acid-like oxidation products obtained from the action of HNO<sub>3</sub> on bituminous coal may be analysed polarographically by means of the two main reduction waves given at the dropping mercury electrode. The first waves at  $E_{\frac{1}{2}}$  of  $\approx -0.5$  V vs. the S.C.E. are probably coalesced waves due to the simultaneous reduction of both nitro and quinone structures, as certain polycyclic quinones and aromatics behave in a like manner when treated with HNO<sub>3</sub>. The second waves at  $E_{\frac{1}{2}}$  of  $\approx -1.5$  V vs. the S.C.E. are believed to be due to the reduction of carbonyl groups, present in the original structure as ketone groups, and as oxanthranol-type structures resulting from the tautomerism of quinol-type reduction products of quinone structures.

G. P. COOK

**1292. Separation of non-ionic surface-active agents from mixtures with anionics by batch ion-exchange.** M. J. Rosen (Brooklyn College, Brooklyn, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1675-1676.—Non-ionic surface-active agents can be separated from mixtures with anionic agents by a batch-exchange procedure in which a solution of the surfactant mixture is stirred with a relatively small amount of a strong anion-exchange resin. The anionic material is adsorbed on the resin, which is removed by filtration. The resin is washed free of non-ionic material with methanol, the non-ionic material is recovered from the filtrate and washings and is determined by refractive-index measurements and ash content. Recoveries averaged  $100 \pm 5\%$ .

G. P. COOK

**1293. Titration method for the quantitative determination of dyes.** E. Bene and K. Géczy (Lab. des Unternehmens für Textilfarbstoffversorgung,

Budapest). *Acta Chim. Acad. Sci. Hung.*, 1957, **11** (1-2), 49-56 (in German).—The method presented is based on the interaction of dyes containing organic anions with certain organic cations to give products that are insoluble in water but soluble in organic solvents. Cetylpyridinium bromide is a suitable cationic material and CHCl<sub>3</sub> is a suitable solvent. The dye, in aq. soln. above a layer of CHCl<sub>3</sub>, is titrated with aq. cetylpyridinium bromide (0.4 g per litre), shaking after each addition; the end-point is shown by decolorisation of the aq. layer. Sodium chloride in concn. < 10 g per litre does not interfere. The end-point is sharp and the precision is better than that of the visual comparator method.

G. BURGER

**1294. Methods of testing cellulose acetate flake.** British Standards Institution (2 Park St., London). B.S. 2880:1957, 13 pp.—The standard covers sampling, and determination of moisture content, ash, acetic acid yield, bulk density, sieve analysis, free acidity, relative viscosity of a 1% soln. at 25°, and viscosity at 25% concn. at 25°.

O. M. WHITTON

**1295. Quantitative chemical analysis of mixtures of protein and non-protein fibres.** British Standards Institution (2 Park St., London). B.S. 2822:1957, 5 pp.—The protein fibre is dissolved from a known dry weight of the mixture by means of alkaline sodium hypochlorite. The residue is collected, washed, dried and weighed; its wt. is corrected, when necessary, for loss in the solvent, and expressed as a percentage of the dry wt. of the mixture. The method is applicable, after the removal of added matter, to binary mixtures of any non-protein fibre (except the bast fibres, the cellulose acetates and Vinyon HH) with one of the following protein fibres—wool, chlorinated wool, raw and degummed silk, raw and bleached tussah silk, mohair, cashmere, Ardil B, Ardil F, Fibrolane BX, Lanital, and soya-bean fibre. If several protein fibres are present, total amounts are obtained but not individual quantities. The method is slightly modified for Vicara.

O. M. WHITTON

**1296. Estimation of nitrogen in poly(vinyl cyanide).** U. Bartels (Inst. of Fibre Tech., Teltow-See, Berlin). *Faserforsch. u. Textiltech.*, 1957, **8** (5), 194-195.—From an investigation of the behaviour of several catalysts in the decomposition of poly(vinyl cyanide) by H<sub>2</sub>SO<sub>4</sub> in the Kjeldahl determination of N, a mixture of K<sub>2</sub>SO<sub>4</sub> and CuSO<sub>4</sub> is found to give high accuracy (within  $\pm 0.22\%$ ), smooth decomposition with no objectionable or poisonous vapours, and sharp end-points. *Procedure*—Conc. H<sub>2</sub>SO<sub>4</sub> (5 ml), K<sub>2</sub>SO<sub>4</sub> (100 mg) and CuSO<sub>4</sub> (100 mg) are added to the sample (60 to 100 mg) in the flask of a Parnar-Wagner apparatus, the flask serving both for the decomposition and the distillation. Decomposition is complete in 2 to 3 hr. In the back-titration of the excess acid, the mixed Tashiro indicator is used (pH 5.2 wine red, pH 5.4 grey-blue, pH 5.6 light green). The method is recommended for general (works) use.

H. L. WHITEHEAD

**1297. Determination of hydroperoxide in methyl methacrylate monomer.** Yuzi Takayama (Mitsubishi Rayon Co. Ltd., Kyobashi, Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1956, **59** (10), 1115-1117.—Methyl methacrylate hydroperoxide (I) oxidises Fe<sup>2+</sup> to Fe<sup>3+</sup>, which are colorimetrically determined with *o*-phenanthroline (II). The presence of peroxides (e.g., benzoyl peroxide), quinol and

acetone does not cause interference. Methanolic soln. of  $\text{HNO}_3$  (1%, v/v) (1 ml),  $\text{H}_3\text{PO}_4$  (6.75 ml in 100 ml of methanol) (0.5 ml), the sample (containing 5 to 40  $\mu\text{g}$  of **I**) and ferrous ammonium sulphate reagent (1 ml) are mixed in the order given and set aside for 15 min. at room temp. (For the preparation of the reagent soln., 2 ml of 0.05 *M* soln. in 1 litre of methanol containing 14 ml of conc.  $\text{H}_2\text{SO}_4$  is mixed with 50 ml of methanol immediately before use.) To the product is added **II** soln. (0.1% in methanol) and the whole is made up to 100 ml with benzene. The extinction is measured at 500  $\mu\text{m}$ . The colour remains unchanged for 30 min. A working curve was prepared by the use of tetralin peroxide as standard. K. SAITO

**1298. Paper chromatography of polyols and dibasic acids, and its application to the analysis of alkyl resins.** A. R. H. Tawn and G. J. May. *J. Oil Col. Chem. Ass.*, 1957, **40**, 528-543.—The resin is saponified with a mixture of alcoholic KOH and benzene, the potassium salts are pptd., filtered off and reserved. The mother liquor is diluted with an equal volume of water, then acidified with HCl, and the fatty acids and unsaponifiable fraction are extracted with light petroleum. The extract is passed consecutively through columns of De-Acidite FF and Zeo-Karb 225, and the polyols in the eluate are recovered by evaporation. Residual free acids retained on the De-Acidite column are recovered with HCl and ethanol; the bulk of the fatty acids, obtained earlier as the potassium salts, are recovered by passage of the salts in acetone soln. through the Zeo-Karb-225 column. Chromatograms are run with ethanol-water-conc. aq.  $\text{NH}_3$  (80:16:4, by vol.) as solvent, on Whatman No. 1 paper, 20-cm vertical strip, the strips being used in sets of 5. Good separation of dibasic acids is obtained with the mixed solvent mentioned, and also with benzene-acetic acid-water (4:4:2, by vol.). Polyols are best separated with *n*-butanol saturated with 1.5 *N* aq.  $\text{NH}_3$ . For the detection of acids, bromocresol purple is used; a mixture of equal vol. of 4%  $\text{Na}_2\text{CO}_3$  soln. and 2%  $\text{KMnO}_4$  soln. shows up unsaturated acids as yellow spots; acids that do not reduce permanganate are shown as wine-red areas. Polyols are indicated by alkaline  $\text{KMnO}_4$  soln. (yellow spots); those containing hydroxyl groups on adjacent carbon atoms are indicated by 1% Pb tetra-acetate soln. in benzene; the presence of dipentaerythritol is demonstrated by means of a soln. of 40 g of ceric ammonium sulphate in 100 ml of 2 *N*  $\text{HNO}_3$ . For recording results, phthalic acid and glycerol are used as reference standards for  $R_F$  values. J. L. PROSSER

**1299. Infra-red spectrum of linseed oil. Stand oils, alkyl resins and modified oils.** J. P. Helme and J. Molines (Ét. Robbe Fr., France). *Peint.-Pigm.-Vern.*, 1957, **33** (6), 524-543.—Characteristic bands that occur in the i.r. spectrum of linseed oil and their changes on modification or polymerisation of the oil are indicated. L. A. O'NEILL

**1300. Determination of the acid value of linseed oils extracted from paints.** L. J. David (S.N.C.F. Laboratories, France). *Peint.-Pigm.-Vern.*, 1957, **33** (7), 629-630.—The oil is extracted by thinning with ether, centrifuging and distillation of the solvent. The oil (3 to 5 g) is then dissolved in ethanol-benzene (3:1) and metal soaps are pptd. by treatment with 0.1 *N* oxalic acid in ethanol for 1 hr. at reflux temp. The mixture is then titrated hot with 0.1 *N* ethanolic KOH to phenolphthalein.

A blank determination is carried out to allow for the oxalic acid, subtracted from the titration, and the acid val. is calculated in the usual way.

L. A. O'NEILL

**1301. Ultra-violet photometric methods of tannin estimation in relation to wattle-extract utilisation.** D. G. Roux (Leather Ind. Res. Inst., Grahamstown, S. Africa). *J. Amer. Leath. Chem. Ass.*, 1957, **52** (6), 319-329.—A u.v. method for estimating the polyphenols of wattle is based on the fact that wattle tannins have a very strong absorption band at  $\lambda_{\text{max.}} = 203 \text{ m}\mu$ , which is readily recorded by a Beckman Model DK-1. For routine analysis it is found that the Unicam SP.500 is satisfactory and a standard curve may be prepared by using it. The relationship of the new method with the hide powder and existing photometric procedures ( $\lambda_{\text{max.}} = 280 \text{ m}\mu$ ) is assessed. C. A. SLATER

**1302. Paper-chromatographic analysis of the naphthenic syntans.** A. N. Roseira (Nat. Inst. of Technol., Rio de Janeiro, Brazil). *J. Amer. Leath. Chem. Ass.*, 1957, **52** (6), 330-335.—The syntans are spotted on to a Schleicher & Schull No. 2043a paper, which is arranged in the form of a cylinder and placed in a solvent system of butanol-ethanol-formic acid-water (8:2:4:5) for development of the chromatogram. The naphthalene syntans are detected by their reaction with basic dyes, and for this purpose methyl violet 2B was found to be most satisfactory. Examples are given of chromatograms of commercial syntans, and a possible application of the method for control during the manufacture of syntans is suggested. C. A. SLATER

**1303. A tentative simplified method for the analysis of ligninsulphonates.** P. A. Deutsch and W. R. Kenzie (Ontario Res. Foundation, Toronto, Canada). *J. Amer. Leath. Chem. Ass.*, 1957, **52** (6), 312-318.—An alternative method to the ALCA Method for tannin analysis, which is suitable for the analysis of ligninsulphonates, is described. *Procedure*—A pickled 2-oz middle split of cowhide is cut into 1-cm squares and de-pickled with  $\text{NaHCO}_3$  and NaCl. It is washed, adjusted to pH 4.8 with *N*  $\text{H}_2\text{SO}_4$  and washed thoroughly with water. It is then dried successively with acetone, acetone-xylene (1:1), and xylene. The hide is dried and ground in a Wiley Mill and the powder produced is used for mixing with the tanning soln. One 15-ml aliquot of the tanning soln. (approx. 2%) is used for determining total solids, and a further 15-ml aliquot is added to the hide powder (7 g) and mixed to a paste. After being set aside for 30 min. the paste is mixed with water (100 ml) and the mixture is left for 10 min. The analysis is then completed as in the ALCA method, the non-tannins soln. being separated from the hide powder, and its total solids being determined. C. A. SLATER

**1304. Rapid determination of zinc, calcium and magnesium in rubber vulcanisates.** C. M. Robertson (Min. Supply, Kidbrooke, London). *I.R.I. Trans.*, 1957, **33** (3), 197-100.—The vulcanisate is dry-ashed at  $> 600^\circ$ , or, if the sample contains chlorine, then wet oxidation with a mixture of  $\text{H}_2\text{SO}_4$  and  $\text{HNO}_3$  should be used. The ash is dissolved in HCl, and metals of the silver, copper and iron groups are removed by the usual methods. Aliquots of the resulting filtrate are titrated with EDTA (disodium salt), (a) at pH 10, with Eriochrome black T as indicator, to give the sum of Zn, Ca and Mg;



(b) after the addition of KCN to complex Zn, and with Eriochrome black T, to give the sum of Ca and Mg; and (c) after the addition of KCN, at pH 12, with murexide as indicator, to give Ca alone. Any soluble barium, which would interfere with the determination of Ca, should be removed from the ash by sulphation before the addition of HCl.

J. L. PROSSER

**1305. Quantitative determination of antioxidants, after a chromatographic separation on completely acetylated filter-paper. I. Determination of phenyl-1-naphthylamine and phenyl-2-naphthylamine.** J. W. H. Zipp (Rubber Res. Inst. T.N.O., Delft), *Rec. Trav. Chim. Pays-Bas*, 1957, **76** (4), 313-316 (in English).—Antioxidants in rubber are extracted by benzene or ethanol-benzene and separated by partition chromatography on acetylated filter-paper. Phenyl-1-naphthylamine and phenyl-2-naphthylamine are determined colorimetrically after being coupled with diazotised sulphanilic acid.

**II. Determination of some p-phenylenediamine derivatives.** J. W. H. Zipp. *Ibid.*, 1957, **76** (4), 317-320.—The antioxidants NN'-diphenyl-, NN'-di-2-naphthyl- and N'-cyclohexyl-N-phenyl-p-phenylenediamine are similarly extracted, separated and oxidised with benzoyl peroxide to the p-quinonedi-imines, which are determined colorimetrically.

M. DAVIS

See also Abstracts 1142, 1203, 1239, 1242, 1352.

#### 4.—BIOCHEMISTRY INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

##### Blood, Bile, Urine, etc.

**1306. Comparative microchemical investigations of the iodine content of [human] blood serum and tear fluid.** A. Pillat, F. Trichtel and M. Oberhummer (Univ.-Augenklinik, Vienna). *Mikrochim. Acta*, 1957, (3-4), 520-526 (in German).—Use was made of the method of Spitzzy and Reese (private communication, publication to follow), by which as little as 0.002  $\mu$ g of iodine can be determined. Tear fluid (0.2 ml) or blood serum (0.5 ml) is ashed with  $\text{CrO}_3$ - $\text{H}_2\text{SO}_4$ . The iodine is thus oxidised to non-volatile  $\text{HIO}_3$ , which is reduced with  $\text{H}_3\text{PO}_3$  and isolated by isothermal diffusion. The final determination is effected by the catalytic action of the iodine on the  $\text{Ce}(\text{SO}_4)_2$ - $\text{H}_2\text{AsO}_3$  system.

L. S. ADLER

**1307. Enzymic method for the estimation of glycerol [in blood].** O. Wieland (II Med. Klinik, Univ. Munich). *Biochem. Z.*, 1957, **329** (4), 313-319.—The estimation of glycerol is based on its phosphorylation to L- $\alpha$ -glycerophosphate with subsequent conversion of this compound into dihydroxyacetone phosphate by DPN; the velocity of DPNH formation is related to the amount of glycerol. Studies of the kinetics of the reactions are reported. The estimation in blood is carried out as follows. The protein-free filtrate of fluoride-plasma (1 ml) is mixed with hydrazine buffer (pH 9.8) (2 ml), 0.02 M DPN (0.08 ml), 0.075 M ATP (0.05 ml) and glycerophosphate dehydrogenase (0.03 ml). After measurement of the extinction at 366  $\mu$  against a control of buffer and DPN the reaction is started by the addition of glycerokinase

(0.01 to 0.02 ml, approx. 500 units). The reaction is followed over a period of up to 50 min. until the  $E_{366}$  reaches a max. value. A linear relationship was established between  $\Delta E_{366}$  and the concentration of glycerol over the range of 0.05 to 0.20  $\mu$ mole per ml. The recovery of added glycerol within this range was 96.8 to 100% in four tests. Application of this method to the estimation of glycerol and L- $\alpha$ -glycerophosphate in rat tissues is described.

G. W. CAMBRIDGE

**1308. Micro-determination of hippuric acid in urine.** H. C. Elliott, jun. (Univ. of Alabama Med. Coll., Birmingham, U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1712-1715.—Hippuric acid is separated from the creatinine and amino acids by ion exchange on Dowex 50-X8 resin. The hippuric acid content of the eluate is determined by differential absorption measurements at 232 and 287  $\mu$  to eliminate interference by uric acid. Recoveries of added hippuric acid to urine were 98.8 to 101.3% over the concn. range of 0.76 to 2.8 mg per ml. Good agreement with another method was obtained.

G. P. COOK

**1309. Quantitative determination of carbohydrates as osazones. (Application of the method to collagen and procollagen.)** W. Grassmann, H. Hörmann and R. Hafter (Max-Planck-Inst. für Eiweiss und Lederforschung, München). *Hoppe-Seyl. Z.*, 1957, **307** (2-6), 87-96.—The optimum conditions for osazone preparation in aq. soln. are a pH of 4 to 6 and a gross excess of phenylhydrazine (I). Coloured by-products lessen the accuracy of the colorimetric reading, but the incorporation of  $\text{NaHSO}_3$  in the reaction mixture prevents their formation. Osazones are formed by N-glycosides and isoglycosides, but not by O-glycosides. Collagen and procollagen, after being freed from non-collagen protein by treatment with trypsin, are heated first with N HCl and then for 2 hr. at 100° with I in the presence of acetate and  $\text{NaHSO}_3$ . The extinction of the osazone is read in the region of 400  $\mu$  where I has little absorption.

F. POWELL

**1310. Separation and identification of inositol phosphoric acid esters by paper chromatography.** J. Schormüller and G. Würdig (Inst. für Lebensmittelchemie und Lebensmitteltechnologie, Tech. Univ., Berlin). *Z. Lebensmittelforsch.*, 1957, **105** (5), 397-403.—The separation of a mixture of inositol phosphoric acids by circular chromatography is described. The flow soln. contains acetone, EDTA (disodium salt) and trichloroacetic acid. The chromatogram is dried in air and sprayed with an aq. soln. containing ammonium molybdate, HCl and  $\text{HClO}_4$ . With u.v. light the substances appear as blue spots on a white ground. The esters are characterised by their  $R_f$  values [the distance (cm) travelled by the substance under test/distance travelled by a control (inositol hexaphosphoric acid or orthophosphoric acid)]. Reproducible results are obtained.

S.C.I. ABSTR.

**1311. Modification of the periodide method for the determination of choline.** G. Smits (Amsterdam Univ., Netherlands). *Biochim. Biophys. Acta*, 1957, **26** (2), 424-427.—Choline periodide, pptd. by adding  $\text{KI}_2$  to a choline solution, is dissolved in 1:2-dichloroethane containing iodine in the presence of water; the extinction of the solvent layer is then a measure of the choline present. *Reagents*—(i) Dissolve 15.7 g of iodine and 20 g of KI in water to 100 ml. Use within 24 hr. (ii) Dilute 5 ml of

Teepol to 100 ml with water. (iii) Dissolve 0.5 g of iodine in 1:2-dichloroethane to 100 ml. Use within 24 hr. *Procedure*—To 2 ml of aqueous choline (5 to 75  $\mu$ g) soln. at 0° to 4° in a stoppered pointed centrifuge tube add 1 drop of (ii) and 0.4 ml of (i) at 0° to 4°. Mix, allow to stand for 30 min. and then centrifuge for 15 min., both at 0° to 4°. Remove the supernatant liquid, add 4 ml of (iii) and dissolve the ppt. by stirring. Add 1 ml of water and shake vigorously for 40 sec. Shake similarly at 0.5-hr. intervals during 1.5 hr. at room temp., then allow to stand till clear. Remove the organic solvent and measure its extinction at 365  $m\mu$  against a blank prepared by shaking 4 ml of (iii) with 1 ml of water. W. H. C. SHAW

**1312. Infra-red spectrographic study of amino acids in a drop of aqueous solution.** C. Duval (Lab. Rech. Micro-anal., E.N.S.C.P., 11 rue Pierre Curie, Paris). *Mikrochim. Acta*, 1957, (3-4), 326-333 (in French).—The i.r. spectra of 18 amino acids have been studied in neutral, acid and alkaline solution. The key bands for the individual acids and those for some simple binary mixtures are given.

L. S. ADLER

**1313. Detection of amino acids in chromatographic analysis.** J. Opieńska-Blauch (Inst. of Physiol. Chem., Med. Acad., Lublin). *Chem. Anal.*, Warsaw, 1957, 2 (2), 123-137.—The behaviour of 28 amino acids in paper-chromatographic separation and their detection with ninhydrin are described. It is recommended that the paper chromatograms be developed with ninhydrin or alloxan at room-temp. as this permits the observation of intermediate colours and allows differentiation of amino acids according to the speed of spot formation.

K. F. SPOREK

**1314. Quantitative determination of amino acids by paper chromatography.** C. A. Porter, D. Margolis and P. Sharp (Boyce Thompson Inst. for Plant Res. Inc., Yonkers 3, New York). *Contr. Boyce Thompson Inst.*, 1957, 18 (11), 465-476.—A method for the estimation of amino acids and amides, separated on paper chromatograms, is described. The papers are sprayed with ninhydrin soln. containing 8-hydroxyquinoline and then heated to 60° in an ethanol atm. for 30 min. The coloured products are eluted with 50% ethanol containing 0.025 *M* phosphate buffer at a pH of 7.0, and determined photometrically.

C. A. SLATER

**1315. Analytical applications of Dutoit's thermovolumetry. Estimation of amino acids.** K. K. Chatterjee and A. K. Ghosh (Dept. of Phys. Chem., Univ. Coll. of Sci. and Tech., Calcutta). *J. Indian Chem. Soc.*, 1957, 34 (5), 407-412.—Some biologically important amino acids have been estimated by the thermometric method of Dutoit (*J. Chim. Phys.*, 1922, 19, 324-331) both individually and in mixtures. The method is useful in that it is rapid, but it is non-specific. Results are accurate to within  $\pm 2\%$ .

C. A. SLATER

**1316. New specific reagent for methionine and its use in paper chromatography.** V. Blazsek (Med. Univ., Targu-Mures, Romania). *Naturwissenschaften*, 1957, 44 (5), 114.—On being sprayed with diazotised 1-naphthylamine reagent (100 mg of 1-naphthylamine dissolved in 10% HCl and then mixed with an equal vol. of 0.5%  $\text{NaNO}_2$  soln.), methionine gives an orange spot, which fluoresces dark red in u.v. light. The sprayed chromatograms are heated

to 80° for 5 min. for colour development. Of all other amino acids, only tryptophan gives a colour with this reagent, but it differs from that given by methionine in being blue-grey, fluorescing yellow in u.v. light. Other sulphur-containing amino acids give no reaction.

E. KAWERAU

**1317. Micro-determination of tyrosine.** P. E. Wenger, D. Monnier and J. Vogel (Lab. Chim. Minérale, Chim. Anal. et Microchim., Univ. Geneva). *Mikrochim. Acta*, 1957, (3-4), 405-416 (in French).—The method described is rapid and, apart from the use of a polarograph, requires no material or device other than those in common laboratory use. The tyrosine is nitrated by the action of hot 0.15 *N*  $\text{HNO}_3$ . The nitrated derivative, which is stable and can be stored, is then polarographically reduced, giving a curve which is specific and reproducible. For 20 to 100- $\mu$ g samples the maximum error is  $\pm 5\%$ ; for 10 to 20- $\mu$ g samples it is  $\pm 7\%$ . Interference in the nitration by -SH groups when cysteine is present in amino-acid mixtures can be eliminated by pptn. with Hg acetate in 0.15 *N*  $\text{HNO}_3$ .

L. S. ADLER

**1318. Agar electrophoresis. II. Quantitative evaluation of agar electrophoresis patterns of human serum proteins by densitometry and comparison with moving boundary electrophoresis.** K. V. Giri, Ravindra Nath and H. Srikanthiah (Biochem. Dept., Indian Inst. of Sci., Bangalore). *J. Indian Inst. Sci.*, A, 1957, 39 (3), 224-236.—A comparative study is made and the advantages of agar electrophoresis over paper electrophoresis as a quant. clinical method for serum protein analysis are discussed.

I. JONES

**1319. Voltage and temperature relationships in paper electrophoresis of serum proteins.** P. G. Moinat and E. F. Tuller (New England Deaconess Hospital, Boston, Mass., U.S.A.). *Anal. Chem.*, 1957, 29 (11), 1655-1658.—Precise experimental conditions for certain variables in paper electrophoresis have been determined. A good separation of the five principal protein fractions is obtained by using the horizontal open-strip method for 15 hr. at 4.9 V per cm at 4°. The principles discussed should be valid for other basic paper-electrophoresis techniques, e.g., the compressed and the hanging-strip methods.

G. P. COOK

**1320. Weight determination of proteins by thermocoagulation.** P. Fleury and R. Eberhard (Lab. de Chim. Biol., Fac. de Pharm., Paris). *Ann. Pharm. Franç.*, 1957, 15, 81-89.—The determination of protein wt. by thermocoagulation is discussed as the reference method. The results obtained by this method in aq. and alcoholic media are compared. The method in aq. media is shown to involve a certain retention of salts by the protein and also a peptisation of the coagulated protein, both of which introduce small negative errors. A new technique for aq. media is described which minimises such errors. It is also shown that the more rapid technique in alcoholic media gives accurate results.

S.C.I. ABSTR.

**1321. Chloric acid method for determining protein-bound iodine by use of iodine-131.** J. F. Goodwin, R. B. Hahn and A. J. Boyle (Wayne State Univ., Detroit, Mich., U.S.A.). *Anal. Chem.*, 1957, 29 (11), 1681-1684.—By using radioactive tracer methods with  $^{131}\text{I}$  it was found that the presence of chromate is not necessary in the chloric acid method

for determining protein-bound iodine, and that, if the digestion is continued to complete dryness, the iodine in a sample of serum is almost wholly lost even when chromate is present. Protein-bound iodine can be separated from inorganic iodine by using either  $\text{HClO}_4$  or trichloroacetic acid as a protein precipitant. G. P. COOK

**1322. Purification of urinary 17-oxosteroid extracts for infra-red analysis.** W. R. Slaunwhite, jun., and L. Neely (Roswell Park Memorial Inst., Buffalo, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1614-1616.—In methods commonly used for preparing neutral urinary extracts, contaminating materials make it difficult to identify i.r. spectra of 17-oxosteroids. Substantial purification can be achieved by washing the dichloromethane soln. of the extract with 0.1 N NaOH and then with 0.1 N HCl, followed by treatment with vegetable charcoal for a few minutes at room temp. This treatment, in conjunction with chromatographic separation, gives i.r. spectra nearly identical with those of standards. G. P. COOK

**1323. Colorimetric assay for cortisone, hydrocortisone and related steroids.** E. P. Schulz and J. D. Neuss (Merck & Co. Inc., Rahway, N.J., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1662-1665.—The method is based on the chromogen produced by the reaction of the steroid with 2:6-di-*tert*-butyl-*p*-cresol in alkaline soln. Cortisone produces a yellow-brown colour with an absorption max. at 471 m $\mu$ , and hydrocortisone develops a blue colour with a max. at 625 m $\mu$ . The best concn. range is from 200 to 500  $\mu\text{g}$ , slightly low results being obtained at the 100- and 600- $\mu\text{g}$  levels. Good results were obtained from various steroid formulations. G. P. COOK

**1324. Chemical method for the determination of urinary aldosterone.** W. Nowaczynski, E. Koiw, J. Genest, R. Tellier, I. Morin, A. Laflamme and P. Robinson (Hotel-Dieu Hospital, Montreal, Quebec). *Canad. J. Biochem. Physiol.*, 1957, **35** (6), 425-443.—The purification of crude neutral extracts of urine for the isolation and determination of aldosterone is described. The crude neutral extracts are successively purified by (i) chromatography on a silica-gel column; (ii) paper-chromatographic separation in ethanediol-toluene; (iii) paper-chromatographic separation in *tert*-butyl alcohol-water-isooctane (25:45:50); and (iv) paper-chromatographic separation in 55% aq. methanol-benzene. Confirmation that the substance isolated is aldosterone is based on the mobilities in the three chromatographic systems used, on the typical u.v. absorption curve, on the satisfactory agreement between the values obtained by u.v. absorption and by the blue tetrazolium reaction, and by the typical chromogen spectra in conc.  $\text{H}_2\text{SO}_4$  and in 100%  $\text{H}_3\text{PO}_4$ . I. JONES

**1325. Measurement of amylolytic activity. Group B. I. Chemical methods.** H. Wildner and G. Wildner (Ireks Forschinst. f. Gärungswiss., Kulmbach, Germany). *Brauwissenschaft*, 1957, **10** (8), 205-210; (9), 233-239.—Detailed descriptions embodying the authors' experience are given of published methods, which include that of Sherman, Kendall and Taylor for the determination of amylase, and the methods of Bang, Baker and Hulton, Euler and Svanberg, and Windisch and Kolbach for the determination of diastatic activity. Published methods for the determination of amylolytic activity in pancreas, kidney and liver are described. P. S. ARUP

lytic activity in pancreas, kidney and liver are described. P. S. ARUP

**1326. A new precipitating reagent for ribonucleic acid and its application to the determination of ribonuclease.** W. Fiers and J. Stockx (Lab. Physiol. Chem., Med. Fak. Univ., Ghent, Belgium). *Naturwissenschaften*, 1957, **44** (5), 115.—To prepare the reagent (I), 10 ml of isopropyl alcohol and 5 ml of 83.5% (w/v) aq.  $\text{Ba}(\text{ClO}_4)_2$  soln. are made up to 100 ml with 96% ethanol. One volume of a soln. containing ribonucleic acid is added to 5 vol. of I. Polynucleotides and proteins are pptd., and enzymic fission products remain in soln. and can be determined spectrophotometrically. Adenylic acid and cytidylic acid are not pptd. under these conditions. This new reagent is compared with the commonly used uranyl acetate -  $\text{HClO}_4$  and ethanol-acetate buffer reagents and the disadvantages of these are discussed. The determination of ribonuclease is briefly described. E. KAWERAU

See also Abstracts 1267, 1412, 1413.

## Drugs

**1327. Quality control of pharmaceuticals. Application of quantitative paper chromatography in conjunction with instrumental methods.** H. J. Pazdera, W. H. McMullen, L. L. Ciacco, S. R. Missan and T. C. Grenfell (Chas. Pfizer & Co., Brooklyn, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1649-1654.—Paper chromatography has been used with u.v. and i.r. spectrophotometry, colorimetry, non-aqueous titrimetry and polarography to give quant. procedures suitable for quality control. A study of precision for sets of three papers showed a variance of from  $\pm 0.5$  to  $\pm 2.4\%$ , and amounts of up to 10 mg have been separated to determine small amounts of impurities. Detailed methods are given for the determination of hydrocortisone, reserpine, hydroxyzine hydrochloride, hydroxydione sodium succinate and carbapentane citrate. G. P. COOK

**1328. Use of paper chromatography with opium alkaloids and morphine derivatives.** A. A. A. Rahman (Pharm. Fac., Univ., Cairo). *Arch. Pharm., Berlin*, 1957, **290**, 321-325.—The  $R_F$  values of morphine, codeine, ethylmorphine, diacetylmorphine, narcotine, papaverine, thebaine, dihydrocodeinone bitartrate, dihydromorphinone hydrochloride, and dihydrohydroxycodeinone hydrochloride were determined with a mixture of butanol, acetic acid and water, or a mixture of butanol, HCl and water as solvent. M. H. SAWISTOWSKI

**1329. Separation of components of tincture of opium and of ampoules of Pantopon and Spasmalgin by means of paper chromatography.** A. A. A. Rahman (Pharm. Fac., Univ., Cairo). *Arch. Pharm., Berlin*, 1957, **290**, 326-329.—The  $R_F$  values of these substances are given and their chromatograms discussed. Separation was effected by a mixture of either butanol, acetic acid and water, or butanol, HCl and water. M. H. SAWISTOWSKI

**1330. Chromatographic purity test of ephedrine hydrochloride. VI. Behaviour of solutions of alkaloidal salts in alumina columns.** H. Böhme and H. Hocke (Pharm.-Chem. Inst., Univ. Marburg-am-Lahn, Germany). *Arch. Pharm., Berlin*, 1957,

290, 422-432.—Ethanol and isopropyl alcohol with various water contents were used as solvents. Ephedrine, being a strong base, is found to a greater extent in solvents containing less water, but at least 10% of water is necessary for complete elution. The columns must be comparatively long as the anion is adsorbed with some difficulty (15 g of alumina is needed for complete isolation of 0.1 g of ephedrine). M. H. SAWISTOWSKI

1331. A new method of assay of *Rauwolfia serpentina*. Bithika Bose (Lady Brabourne Coll., Calcutta). *J. Instn Chem. India*, 1957, **29** (3), 166.—In the assay method described for total alkaloids in *Rauwolfia serpentina*, powdered root (5 g) was shaken with conc. aq.  $\text{NH}_3$  (5 ml) and then with a mixture (45 ml) of equal vol. of  $\text{CHCl}_3$  and methanol and set aside overnight. The filtrate (25 ml) was extracted with 6% (v/v) acetic acid (25-, 20-, 15- and 10-ml portions). The combined acid extracts were extracted with 10 ml of  $\text{CHCl}_3$ , washed with water, and added to the main extract. The residue was free from alkaloids. The acetic acid extract was filtered, rendered alkaline with 10 ml of conc. aq.  $\text{NH}_3$  and extracted with 25-, 20-, 15- and 10-ml portions of *n*-butanol. The total *n*-butanol extract was washed twice with water, the water was separated by adding  $\text{CHCl}_3$  (10 ml) and drawn off. The extract was filtered, evaporated, dried and weighed. The residue was repeatedly evaporated with addition of ethanol to constant wt. Four different samples were found to contain 3.48 to 4.20% of total alkaloids by this assay, but only 0.95 to 1.32% by the B.P.C. method. O. M. WHITTON

1332. Determination of the alkaloids in galenical preparations with Tropaeolin OO. B. Schmitz and W. Menges (Andraee-Noris Zahn A.-G., Frankfurt/M., Germany). *Dtsch. ApothZtg*, 1957, **97** (34), 747-749.—A colorimetric method is described for the determination of strychnine and quinine alkaloids in tinctures with Tropaeolin OO. About 0.5 g of tincture of cinchona or 1 g of the compound tincture (D.A.B. 6) is diluted to 250 ml with water, and a 5-ml portion is mixed with 10 ml of an acetate buffer soln. of pH 4.6 and 3 ml of a satd. aq. Tropaeolin OO soln. The well-shaken mixture is then extracted with  $\text{CHCl}_3$  ( $4 \times 5$  ml), and the combined extracts are acidified with 3 ml of an acid reagent (1 ml of conc.  $\text{H}_2\text{SO}_4$  and 99 ml of methanol), and made up to 50 ml with  $\text{CHCl}_3$ . The extinction is then determined and the alkaloid content estimated by comparisons with standard curves. Blank determinations are necessary. Tincture of *nux vomica* may be similarly assayed. G. R. WHALLEY

1333. Microchemical determination of nicotine in tobacco. G. Gorbach and H. Kögler (Inst. Biochem. Technol., Graz, Austria). *Mikrochim. Acta*, 1957, (3-4), 572-576 (in German).—The direct acidimetric titration of nicotine is unsuited for microchemical application, owing to its diffuse end-point. The indirect method described attains, on the micro scale, the accuracy of the macro-determination. The nicotine is distilled or steam-distilled from alkaline soln., and the distillate is neutralised with 0.1 N HCl to methyl red. The nicotine is then pptd. as the monopicrate by the addition of a known excess of 1% picric acid soln. Quantitative and crystalline pptn. occurs after 2 to 3 hr. in a refrigerator. The ppt. is filtered off and the excess of picric acid in the filtrate and washings is determined iodimetrically. L. S. ADLER

1334. Identification of glycosides, furanochromones, alkaloids and their salts by the method of determination of eutectic fusion. M. Kh. Gluzman and V. P. Rubtsova (Kharkov Sci. Res. Chem.-Pharm. Inst.). *Aptekhn. Delo*, 1957, **6** (1), 10-14.—The method of identifying organic compounds by means of eutectic melting-points (*Zhur. Prikl. Khim.*, 1953, **26**, 1223) is applied to glycosides, furanochromones, alkaloids and alkaloidal salts which either melt with decomposition or melt at inconveniently high temperatures. Eutectic melting-points are given for over 130 binary systems formed by 31 compounds of the type under examination. E. HAYES

1335. Preliminary phytochemical investigation of *Digitalis lutea* of second-year growth. S. M. Schwartz (Univ. of Minnesota, St. Paul, U.S.A.). *Dissert. Abstr.*, 1957, **17** (5), 988.—In the paper-chromatographic analysis of cardiac glycosides, PbO is unsatisfactory for clarification of aq. extracts of *D. lutea* leaves as it adsorbs and deacetylates the glycosides. Four (unspecified) solvent systems were used, all containing formamide. Three separated the glycosides *per se*, and one separated digoxigenin and digitoxigenin. Separation was also possible on a formamide-impregnated cellulose column, eluting with benzene- $\text{CHCl}_3$  (6:4). By partitioning between this solvent mixture and methanol, separation of  $\alpha$ - and  $\beta$ -acetyldigoxin was effected. S.C.I. ABSTR.

1336. Anthraquinones. IX. Calibration curves for determination of anthraquinones. H. Auerhoff (Pharm.-Chem. Inst., Tübingen Univ.). *Arch. Pharm., Berlin*, 1957, **290**; *Mitt. dtsch. pharm. Ges.*, 1957, **27** (6), 81-82.—The method of preparing calibration curves by measuring the extinction of alkaline soln. of 1:8-dihydroxyanthraquinones is criticised. These solutions are very unstable and their colour depends on the method of preparation. More suitable for calibration purposes are 1% soln. of  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ . M. H. SAWISTOWSKI

1337. Determination of capsaicin. P. Spanyol, E. Kevei and M. Kiszal (Forschungsinst. für Konserv-, Fleisch- und Kälte-ind., Budapest). *Acta Chim. Acad. Sci. Hung.*, 1957, **11** (1-2), 137-149 (in German).—Diazotised sulphanilic acid in alkaline ethanol soln. gives a specific colour reaction with capsaicin. A procedure is presented for the extraction of capsaicin and its determination. Prescribed conditions must be closely adhered to. A precision of 10% is attainable. G. BURGER

1338. Identification of antibiotics as tetraphenylboron derivatives. M. Zeif, R. Woodside and E. Huber (J. T. Baker Chem. Co., Phillipsburg, N.J., U.S.A.). *Antibiot. & Chemother.*, 1957, **7** (11), 604-605.—Crystalline derivatives, suitable for identification purposes, are obtained by reaction of sodium tetraphenylboron with erythromycin, carbomycin, leucomycin and oleandomycin. Penicillin, streptomycin and six other antibiotics do not react. W. H. C. SHAW

1339. Fluorimetric determination of chlortetracycline. D. H. Feldman, H. S. Kelsey and J. C. Cavagnol (American Cyanamid Co., Pearl River, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1697-1700.—The method is based on the alkaline degradation of chlortetracycline (I) to isochlortetracycline at pH 7.5 and in the absence of dissolved air. The fluorescence is directly proportional to the concn. of



**I** before degradation. Variables studied included the sensitivity of fluorescence intensity to pH, the choice of secondary filters, and the effect of organic solvents. Routine determinations on fermentation-broth samples gave a precision of  $\pm 2.2\%$  (95% confidence limits). G. P. COOK

**1340. Differential spectrophotometric determination of novobiocin.** P. Sensi, G. G. Gallo and L. Chiesa (Lepetit S.p.A., Milan, Italy). *Anal. Chem.*, 1957, **29** (11), 1611-1613.—Novobiocin and dihydronovobiocin are determined in their mixtures by differential spectrophotometric measurement of their acid hydrolysis products at 250 and 330 m $\mu$ . Beer-Lambert's law is followed in the concn. range 2 to 12  $\mu$ g per ml. Recoveries of both compounds exceed 95%. G. P. COOK

**1341. Isolation and properties of the components of staphylomycin.** H. Vanderhaeghe, P. Van Dijk, G. Parmentier and P. De Somer. (Rega Inst., Catholic Univ., Louvain, Belgium). *Antibiot. & Chemother.*, 1957, **7** (11), 606-614.—The two main synergistic factors of staphylomycin are separated by descending chromatography on Whatman No. 1 paper for 72 hr. in the system propane-1:2-diol-benzene, and then assayed by a bio-autograph technique. Details are also given of the chemical and physical constants of the two factors and of a colorimetric assay with *p*-dimethylaminobenzaldehyde. A third biologically active factor, not isolated in a pure state, is shown to be present. W. H. C. SHAW

**1342. Detection of barbituric acid derivatives.** L. I. Rapoport (Central Pharm. Res. Lab. "GAPU," Min. of Health, UkrSSR). *Apteknoe Delo*, 1957, **6** (1), 17-22.—General and specific tests for the barbiturates described in the literature are critically evaluated. New tests are proposed for amylobarbitone Na, pentobarbitone Na and thiopentone Na. For the detection of pentobarbitone, 0.01 to 0.02 g is dissolved in 10 ml of conc. H<sub>2</sub>SO<sub>4</sub> and 10 drops of a 20% soln. of *p*-dimethylaminobenzaldehyde in conc. H<sub>2</sub>SO<sub>4</sub> are added; the development of a blood-red colour after the mixture has been heated on a water bath for 8 to 10 sec. indicates the presence of pentobarbitone. Under the same conditions, amylobarbitone Na gives a reddish soln. with a green fluorescence after 1 to 2 min. For the detection of thiopentone Na, 0.05 g is dissolved in 1 ml of water and a few drops of a 4% soln. of Cu acetate are added. A greyish-green ppt. is formed which turns yellow on being heated with 3 ml of water; it dissolves in ether to give a yellow soln. With CuSO<sub>4</sub> instead of Cu acetate, the colour of the ppt. does not change when it is shaken with water and it dissolves in ether to form a green soln. E. HAYES

**1343. Identification of local anaesthetics.** M. Brandstätter-Kuhnert and H. Grimm (Pharmacol. Inst., Univ. Innsbruck). *Mikrochim. Acta*, 1957, (3-4), 427-447 (in German).—A scheme of analysis for 28 local anaesthetics has been worked out, and is based on the micro-determination of the melting-points of one or more of the following derivatives—the anthraquinone- $\beta$ -sulphonate, the diluturate, the flavanate, the picrate and the styphnate. The two or more different melting-points of some of the derivatives are attributed mainly to polymorphism. The table incorporating the necessary data includes the figures for 49 pure bases and salts. L. S. ADLER

**1344. Titration of phenazone in non-aqueous solution with colour indicators.** T. Jasinski and I. Pawelczak (Z. Zakl. Chem. Farm., A.M. w Gdańsku). *Acta Polon. Pharm.*, 1957, **14** (2), 91-93.—Phenazone soln. in glacial acetic acid, glacial acetic acid-acetic anhydride (1:1) or acetic anhydride were titrated with 0.1 N or 0.01 N HClO<sub>4</sub>. Methyl violet, crystal violet and malachite green were found to be the most suitable indicators. B. LAKE

**1345. Application of paper chromatography to the separation of sulphonamides in medicinal preparations.** H. Rafałowska, T. Kowalczyk, S. Ochab and B. Paprocka (1st Inst. Med. Drugs, Warsaw). *Chem. Anal.*, Warsaw, 1957, **2** (4), 366-375.—Results are presented, together with some chromatograms, of work undertaken to correlate, simplify and improve the accuracy of techniques described in the literature. On the basis of the experiments the following solvents are recommended—acidic: *n*-butanol-acetic acid-water (100:20:48); alkaline: *n*-butanol-10% aq. NH<sub>3</sub> (100:30); neutral: *n*-butanol-water (100:19). As developer, a 1% soln. of *p*-dimethylaminobenzaldehyde containing 1% of HCl, which gives an immediate bright-yellow coloration, is stated to be the most sensitive. It gives the most permanent chromatograms and is simple to use. T. M. M.

**1346. Quantitative determination of "Étazol" [2-(*p*-aminobenzenesulphonamido)-5-ethyl-1:3:4-thiadiazole] and sulphadimidine.** Ts. I. Shakh (Kiev Post-grad. Med. Inst.). *Apteknoe Delo*, 1957, **6** (1), 22-24.—Étazol and sulphadimidine can be quant. iodinated by ICl or ICl<sub>3</sub> under controlled conditions to form di-iodo derivatives. *Determination of sulphadimidine*—A weighed sample (0.5 to 0.6 g) is dissolved in the minimum amount of HCl, and the soln. is made up to 100 ml. A 10-ml aliquot is transferred to a calibrated flask; 200 ml of hot water and 30 ml of 0.1 N ICl (in HCl) are added, and when cold the soln. is made up to 250 ml with water. The soln. is filtered and the first 10 to 15 ml of filtrate is rejected; 1 g of KI is added to 100 ml of the subsequent filtrate and the liberated iodine is titrated in the usual way. *Determination of Étazol*—A weighed sample (0.5 to 0.6 g) is dissolved in 10 ml of dil. HCl by gentle heat and the soln. is made up to 100 ml with water. To a 10-ml aliquot of this soln. in a stoppered flask are added 300 ml of hot water and 25 ml of 0.1 N ICl; the flask is shaken and set aside for 10 to 15 min., 1 to 2 g of KI is added and the liberated iodine is titrated against 0.1 N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. E. HAYES

**1347. Estimation of mercury in organic compounds.** F. Lupant-André (Univ. Libre, Brussels). *J. Pharm. Belg.*, 1957, **12** (9-10), 423-429.—The soln. of the Hg compound (50 to 100 ml) is mixed with KCN (2% soln.) to pH 10 to 11. The excess of cyanide is neutralised by addition of 2 N H<sub>2</sub>SO<sub>4</sub> to pH 7, 0.1 N H<sub>2</sub>SO<sub>4</sub> to  $\approx$  pH 5.7 and finally 0.01 N H<sub>2</sub>SO<sub>4</sub> to exactly pH 5.5. After adding 20% (w/v) Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>·5H<sub>2</sub>O (5 ml) the alkalinity is titrated with H<sub>2</sub>SO<sub>4</sub> to exactly pH 5.5. D. P. FELIX

**1348. Detection and determination of Mervitin [methylamphetamine].** M. Weiser and M. K. Zacherl (Med.-chem. Inst. Tierärztlichen Hochschule, Vienna). *Mikrochim. Acta*, 1957, (3-4), 577-586 (in German).—Small amounts of methylamphetamine (**I**) can be detected and determined spectrophotometrically. The **I** reacts with Cu

acetate and  $\text{CS}_2$  in the presence of aq.  $\text{NH}_3$  to give the copper salt of methylamphetamine dithiocarbamate. The reaction is a general one for secondary amines. The sample is therefore first tested with formaldehyde soln. -  $\text{H}_2\text{SO}_4$ . A brick-red colour, changing through brown to olive green, indicates the presence of **I** or amphetamine. The  $\text{CHCl}_3$  soln. of the **I**-Cu complex is stable to alkali, while that of the amphetamine complex is not. The two amines may thus be distinguished and, further, **I** may be determined in the presence of considerable amounts of amphetamine. L. S. ADLER

1349. *m*-Aminophenol in aminosalicic acid by the U. S. P. method. W. D. Kumler and I. J. Szekely (School of Pharm., Univ. of Calif., San Francisco, U.S.A.). *Drug Standards*, 1957, **25** (3), 69-72.—Photometric methods are preferred to the U. S. P. XV gravimetric method for the determination of *m*-aminophenol (**I**) in aminosalicic acid (**II**). The U. S. P. procedure over-estimates the amount of **I**, because the residue weighed may contain some **II** and decomposition products of **II**. A. R. ROGERS

1350. Refractometric method for the determination of nikethamide. S. P. Miskidzh'yan (Lvov Med. Inst.). *Apteknoe Delo*, 1957, **6** (1), 48-50.—Tables are given showing the relationship between the refractive index and concn. of aq. soln. of nikethamide at 15°, 18°, 20° and 22° over the concn. range 5 to 30%. In the range of temperature studied, the mean temperature coefficient of the refractive index is 0.00020 per degree. The results are used in the refractometric determination of pharmaceutical soln. of nikethamide. E. HAYES

1351. Volumetric determination of and microchemical differentiation between mono- and dihydrazino-phthalazines. G. C. Sandri (Ist. Chim. Farm. Univ., Ferrara, Italy). *Boll. Chim. Farm.*, 1957, **96** (10), 431-436.—The two compounds 1-hydrazinophthalazine (hydrallazine) and 1:4-dihydrazinophthalazine can be determined by oxidation with 0.1 N periodic acid in HCl soln. which causes a vigorous evolution of  $\text{N}_2$ ; on addition of KI the excess of iodine is titrated with 0.1 N  $\text{Na}_2\text{S}_2\text{O}_3$ , each hydrazine group requiring four equivalents of iodine. The prep. of compounds of the mono-hydrazino derivatives with  $\text{CdCl}_2$  and  $\text{CdBr}_2$  are described and photographs of the well-defined crystals are provided. P. HAAS

1352. Application of Fujiwara's alkaline pyridine reaction to tertiary acetylenic halogenated alcohols. C. Dumont (Lab. de Toxicol. et d'Hygiène Ind., Paris). *Ann. Pharm. Franç.*, 1957, **15** (4), 216-223.—The Fujiwara colour reaction can be applied to 1-bromo- (**I**) and 1-iodo-3-methylpent-1-yn-3-ol, 1-bromo-3-methyl-3-(*p*-nitrobenzoyloxy)pent-1-yne, 3-methyl-3-(2:2-trichloro-1-hydroxyethoxy)pent-1-yne, 1-chloro- and 1-bromo-2-(1-hydroxycyclohex-1-yl)acetylene, and 1-ethynyl-1-(2:2:2-trichloro-1-hydroxyethoxy)cyclohexane, which give red-violet colours with sensitivities of 5  $\mu\text{g}$  for the bromo deriv. and 50 to 100  $\mu\text{g}$  for chloro and iodo deriv. The reaction is not specific for  $-\text{C}\equiv\text{CX}$  ( $\text{X}$  = halogen), and three compounds are listed which only give a yellow colour at a sensitivity of 5 mg. *Procedure*—To 5 ml of distilled pyridine are added 0.1 ml of a pyridine soln. of the compound and 2 ml of 50% aq. NaOH. After vigorous shaking the tube is heated on a boiling-water bath for 10 to 15 min. The pink colour develops in the pyridine

layer. The reaction is applied quant. to **I** which gives an absorption max. at 550  $\text{m}\mu$ , and a linear relation between concn. and extinction is obtained from 5 to 80  $\mu\text{g}$  (in 5 ml of pyridine).

E. J. H. BIRCH

1353. Method of identification and estimation of ethinyloestradiol in pharmaceutical preparations. C. Heusgham and J. M. Jehotte (Univ. Liège, Lab. de Chim. Méd.). *J. Pharm. Belg.*, 1957, **12** (9-10), 418-422.—Ethinyloestradiol may be detected in microgram quantities by the characteristic red colour with green fluorescence that it gives when an aq. suspension is treated with ethanol -  $\text{H}_2\text{SO}_4$  reagent. Certain natural steroids give a yellow or green fluorescence when present in large amounts (10  $\times$  wt.) but no colour. This reaction is quant., the colour being determined in a colorimeter at 535  $\text{m}\mu$ , and can be used to estimate ethinyloestradiol in preparations, e.g., lozenges and tablets. D. P. FELIX

1354. Determination of aqueous sucrose solutions, in particular of Sirupus Simplex D.A.B. 6. L. Stark and H. von Forell (Städt. Rudolf-Virchow-Krankenhaus, Berlin). *Dtsch. ApothZtg*, 1957, **97**, 629-630.—Since the sp. rotation changes with concn. according to the equation  $[\alpha]_D^{20} = 66.462 + 0.0087c - 0.000235c^2$  [ $c$  = sucrose (%)], this should be taken into account when calculating results. M. H. SAWISTOWSKI

1355. Quantitative determination of small concentrations of ethanol in biological materials and pharmaceutical preparations. A. S. Khodak (Forensic Med. Office, Bryansk District, USSR). *Apteknoe Delo*, 1957, **6** (1), 42-45.—Ethanol is removed from a weighed sample of material by distillation on a sand-bath and the distillate is diluted to give a concn. of ethanol  $\geq 10\%$ . The soln. is added drop by drop from a burette to 2 ml of a mixture (1:2) of ethanol and isoamyl alcohol until a cloudiness develops. A similar titration is carried out with water; the difference gives the vol. of ethanol in the test soln. E. HAYES

1356. Nephelometric assay of spirits of peppermint and camphor. S. R. Conte, P. F. Smith and B. J. Sciarrone (Coll. of Pharm., Rutgers Univ., Newark, N.J., U.S.A.). *Drug Standards*, 1957, **25** (4), 105-111.—For the control assay of spirit of peppermint, mix the sample (4 ml) with a 25% aq. soln. of purified sodium lauryl sulphate (2 ml), dilute a 2-ml aliquot with water to 250 ml, mix, and measure in a nephelometer. Calculate with the aid of a calibration curve obtained with a spirit prepared from the same batch of oil. A similar procedure with Polysorbate 80 as dispersing agent is described for the nephelometric assay of spirit of camphor. A. R. ROGERS

1357. Amperometric titration of ammonium sulphate in medicinal sera. M. K. Abramov and I. L. Teodorovich (Tashkent Pharm. Inst.). *Zhur. Anal. Khim.*, 1957, **12** (4), 566.—The sulphate is pptd. with 20 ml of 0.05 M  $\text{Pb}(\text{NO}_3)_2$ , heated in a boiling-water bath for 3 to 5 min., then cooled and mixed with 66 ml of acetone or ethanol, and diluted to 200 ml with water. An aliquot (30 ml) is treated with 0.2 ml of saturated  $\text{KNO}_3$  soln. and the excess of  $\text{Pb}(\text{NO}_3)_2$  is titrated with 0.01 M  $\text{K}_4\text{Fe}(\text{CN})_6$ , with the use of a rotating platinum micro-electrode. G. S. SMITH

See also Abstracts 1208, 1267, 1323.

## Food

1358. Nitrogen-containing non-sugars. II. Method for routine determination of glutamic acid, aspartic acid and  $\gamma$ -aminobutyric acid in sugar-factory products. F. Schneider, E. Reinefeld and H. Müller. *Zucker-Beihfte*, 1957, **3**, 78-85.—An electrophoretic method is described followed by photometric determination after treating with ninhydrin. N. E.

1359. Determination of saponin in granulated sugar; method of H. G. Walker adapted to routine control. R. R. West and R. S. Gaddie. *J. Amer. Soc. Sugar Beet Technol.*, 1956-7, **9**, 146-152.—A modification of Walker's method (cf. *Anal. Abstr.*, 1957, **4**, 3752), suitable for routine control, is described and the results are tabulated.

SUGAR IND. ABSTR.

1360. Determination of water in starch and starch products. E. Eberius and W. Kempf. *Stärke*, 1957, **4**, 77-81.—The application of the Karl Fischer method to starches, starch products and by-products is compared with physical methods. For native granules two variations of the Karl Fischer technique were used, namely, direct titration with the reagent, and preliminary dispersion of the granules in anhydrous methanol. It was found that the former method gave values for the water content which were consistently 0.5% less than values from the latter. The water content obtained by direct titration also corresponded to that given by classical oven-drying methods. It was considered likely that methanol caused some diffusion of water out of the interior of the starch granules. For starch syrups, comparisons were made between the direct titration with Karl Fischer reagent and corrected readings obtained with a refractometer, and values obtained here did not differ by more than 0.08%. E. DUX

1361. Analytical and paper-chromatographic investigation of the sugars of honey and artificial honey. K. Täufel and K. Müller. *Ernährungs-forschung*, 1957, **2**, 70-82.—Analytical figures and tabulated  $R_{\text{mucose}}$  figures are given for the sugars in various genuine and artificial honeys. Chromatograms were developed with butanol-acetic acid-water (4:5:1) for three days at room temp. and sprayed with (a) aniline-diphenylamine-phosphoric acid reagent for all sugars, (b) aniline hydrogen phthalate in butanol for reducing sugars, and (c) resorcinol-trichloroacetic acid for ketose sugars. The chromatographic sugar "spectrum" distinguished the artificial honeys from the genuine samples, and the fructose to glucose ratio was from 106 to 138 for genuine honey and from 92 to 93 for the artificial samples. E. C. APLING

1362. Some physical properties of milk. IV. Maximum density of milk. C. H. Witnab, T. M. Medved and W. D. Rutz (Kansas State Coll., Manhattan, U.S.A.). *J. Dairy Sci.*, 1957, **40** (7), 856-861.—A study is made of the temp. at which max. density of milk is observed in relation to the amount of added water. For pure milk, 10 hr. after pasteurisation, the average temp. of max. density was  $-5.20^{\circ}$ ; this temp. rises linearly with percentage of added water to that of water ( $+4^{\circ}$ ). Measurement of the temp. of max. density is suggested as a method for the determination of added water, and the significance of the observations is discussed. W. H. C. SHAW

1363. Modified Gerber method for estimation of fat in dried milk. W. V. Falkenhahn (Dairy Div., Dept. of Agric., Auckland, N. Zealand). *N. Z. J. Sci. Tech.*, 1957, **38** (6), 571-576.—The Gerber method is modified by introducing a centrifuge heated to  $65^{\circ}$  and a mechanical shaker. The tests can be completed in less than 45 min. The readings are reproducible within  $\pm 0.05\%$  and agree with those by the British Standards Röse-Gottlieb method within  $\pm 0.2\%$ . The British Standards Röse-Gottlieb and the Mojonnier modification are discussed as gravimetric reference methods for standardising the modified Gerber results. R. H. HURST

1364. The fatty acids of butter fat and the volatile acids formed on oxidation. J. C. Hawke (D.S.I.R., Wellington, N. Zealand). *J. Dairy Res.*, 1957, **24** (3), 366-371.—A description is given of the gas-liquid chromatographic methods applied to the steam-volatile fatty acids and to the methyl esters of the non-volatile fatty acids from butter fat. A total analysis of butter fat agreed well with results obtained by ester-fractionation methods. No short-chain fatty acids with odd C numbers were found in the fresh fat, but these were detected after oxidation by air at  $35^{\circ}$  or  $70^{\circ}$  for 6 hr. W. H. C. SHAW

1365. Detection of adulteration of ghee with vanaspati. II. Measurement of turbidity temperature with benzyl alcohol-glycerol as solvent. H. S. R. Desikachar, M. V. Patwardhan, L. V. L. Sastry, M. Srinivasan and V. Subrahmanyam (Centr. Food Tech. Res. Inst., Mysore). *J. Sci. Ind. Res., B, India*, 1957, **16** (5), 216-219.—Turbidity temp. of ghee and vanaspati have been determined in different pairs of solvents. The turbidity temp. of vanaspati is uniformly higher than that of ghee by  $20^{\circ}$  to  $25^{\circ}$ , and this is suggested as a method for detecting vanaspati in ghee. It has been found that a mixture of benzyl alcohol and glycerol (100:17.1, v/w) is a suitable solvent system for turbidity measurements, and levels of 20% adulteration can be detected since such mixtures consistently show a turbidity temp.  $10^{\circ}$  higher than that of pure ghee. Turbidity temp. of some common edible fats are reported. C. A. SLATER

1366. Qualitative and quantitative determination of amino acids in some important edible fungi. C. Seelkopf and H. Schuster (Inst. für Pharmazie u. Lebensmittelchemie, Univ., Würzburg, Germany). *Z. Lebensmittelforsch.*, 1957, **106** (3), 177-187.—The proteins of the fungi can be isolated approx. quant. by the Neuberg and Kerb method (*Biochem. Z.*, 1914, **119**, 498), provided that the pptn. (with the use of aq. 25% mercuric acetate and 10% NaOH) is repeated on the filtrate. The proteins are hydrolysed by heating at  $100^{\circ}$  for 3 hr. in an atmosphere of N with 6 N HCl-5 N formic acid (1:1) with  $\text{Hg}^{2+}$  as catalyst. Special procedures and (known) colorimetric methods are used for the separate determination of tryptophan (which is destroyed on acid hydrolysis) and for that of cystine (destroyed by the  $\text{Hg}^{2+}$  catalyst). The yields of all the other amino acids are approx. quant. The amino acids are detected and quant. determined by paper-chromatographic methods, which are described. Protein contents are given for nine edible fungi. As the fungi contain appreciable amounts of non-protein N, the published data based on the total Kjeldahl-N content are high

(by  $\approx 7\%$ ). With the exception of truffle protein, which contains no methionine, the proteins contain all the essential amino acids. P. S. ARUP

**1367. Determination and characterisation of the phenolic substances of fruit.** C. J. B. Smit (W. P. Fruit Res. Sta., Stellenbosch, S. Africa). *S. Afr. Ind. Chem.*, 1957, **11** (5), 93-95.—Observations made on the effect of pH on the u.v. absorption spectra of catechin and tannic acid showed that both substances exhibit a reversible shift of the u.v. absorption spectrum to higher wavelengths when exposed to highly alkaline conditions. The possibilities of using pH adjustments as a further aid in the characterisation of polyphenols are discussed. S.C.I. ABSTR.

**1368. Determination of total acids in fruit juice and wine.** J. Koch and G. Schiffner (Inst. für Obst und Gemüseverwertung, Geisenheim, Austria). *Z. Lebensmitteluntersuch.*, 1957, **106** (2), 119-122.—Owing to the differences between the curves of NaOH titre vs. pH for citric acid and for the other principal fruit juices, it is proposed that the electrometric titration end-point for citrus juices should be at pH 8.4, and that for other fruit juices or wines at pH 7. By this procedure, correct results are obtained for the acid content of grape juices and wines and for citrus juices, but the acids of black-currant and apple juices will be under-estimated by 0.4 and 0.2 g per litre, respectively. P. S. ARUP

**1369. The determination of caffeine in drugs (tea, coffee) by the method of isotopic dilution.** G. Ciranni, R. Giuliano and M. Zifferero (Inst. Pharm. Toxicol. Chem., Univ., Rome). *Ann. Chim., Roma*, 1957, **47** (6), 646-648.—Theophylline was treated with labelled methyl iodide to give caffeine labelled in the 7-methyl position. After the addition of a known amount of the labelled caffeine, the total caffeine in the drug was determined by the isotopic dilution method. This showed the presence of larger amounts of caffeine than were indicated by the methods commonly employed. A. G. COOPER

**1370. Determination of the proteolytic activity of enzymic preparations used for the stabilisation of beer.** E. Paukner, W. Kleber and M. Lindemann (Wissenschaftl. Stat. f. Brauerei, Munich). *Brau-wissenschaft*, 1957, **10** (8), 204.—To a buffered (at pH 4.6) soln. of gelatin (10% w/v) (40 ml maintained at 40°) add a 1% soln. of the prep. (1 ml) and transfer the mixture to a Höppler viscometer which is maintained at 40°. Determine the efflux-time exactly 7 min. after addition of the prep., repeat the measurement at intervals of 15 min. for 1-25 hr. and plot a curve showing decreases in efflux-times with time, the first measurement being taken as the zero value. By reference to the curve an empirical value for the proteolytic activity of the prep. is calculated. P. S. ARUP

**1371. Determination of volatile acids [in wine].** K. Woidech (Lebensmittelversuchsanstalt, Wien). *Dtsch. LebensmittelRdsch.*, 1956, **52**, 267.—A steam-distillation apparatus is described for the determination of volatile acids, e.g., sulphurous acid, nitrate, nitrite and organic bromo compounds in wine and fruit juices. D. P. FELIX

**1372. New method for the detection of dulcin (N-phenetylurea) in foodstuffs.** F. Bucci and V. Amormino (Ist. Superiore di Sanità, Rome). *Ann.*

*Chim., Roma*, 1957, **47** (7-8), 770-784.—A colorimetric procedure is suggested in which the dulcin is hydrolysed by boiling with dil. HCl to *p*-phenetidine, which is then diazotised and coupled with a phenol or aromatic amine. The method has been applied to wine, beer, preserves, syrups and fruit juices. A. G. COOPER

**1373. Investigation by paper partition chromatography of antiseptics derived from benzoic acid which may be added to food products.** J. L. Joux (Service de Recherches de l'Inst. Appert). *Ann. Falsif.*, 1957, **50**, 205-211.—The substances studied were benzoic, *o*- and *p*-chlorobenzoic, salicylic and *p*-hydroxybenzoic acids, and the methyl, propyl and butyl esters of *p*-hydroxybenzoic acid. The material is boiled under reflux with dil. H<sub>2</sub>SO<sub>4</sub> and filtered, and the acids are extracted from the filtrate with ether-light petroleum (1:1). After being washed, they are transferred back to an alkaline aq. solvent, saponified by boiling under reflux, and, after acidification, the acids are extracted into a small vol. of ether-light petroleum. A preliminary test by circular paper chromatography, with salicylic acid as reference substance, enables *R<sub>F</sub>* and *R<sub>S</sub>* values to be determined, the solvent being isoamyl alcohol-ethanol-1.5 N aq. NH<sub>3</sub> (6:3:2) and the spray reagent an aq. alcoholic soln. of methyl red and bromophenol blue buffered at pH 7.0. Further identification is by means of descending chromatography, with spots of the unknown, of suitable pure acids as decided by the preliminary test, and of mixtures of unknown and pure compounds. The identification is made by comparison of unknown with known, and also by non-separation of one or more of the mixtures. *R<sub>F</sub>* and *R<sub>S</sub>* values are tabulated. The method is not quantitative, and does not distinguish between free *p*-hydroxybenzoic acid and its esters when present together, nor between the esters. The free acid can be found in the extract both before and after saponification, whereas esters will be indicated only after saponification. R. E. ESSERY

**1374. Chromatographic determination of oxidised acids in fats.** M. Burnet and P. Desnuelle (Lab. Nat. des Matières Grasses, Marseilles). *Oléi Min.*, 1957, **34** (7), 321-327.—Limitations in the normal procedures for determining oxidised acids from their insolubility in light petroleum, e.g., by the IUPAC method, are demonstrated. Certain oxidised acids, e.g., monohydroxy and ketohydroxy acids, have significant solubility and the solubility of oxidised acids is in general enhanced by the presence of unoxidised acids. Oxygenated groups, e.g., OH or CO, are found in the light petroleum-soluble fraction of acids from an oxidised fat. A method has been developed for determining oxidised acids based on reversed-phase partition chromatography. Powdered rubber impregnated with light petroleum is used as the stationary phase, acid aq. acetone as the mobile phase. Oxidised acids migrate more rapidly than unoxidised acids, with the exception of lauric and lower acids, and in the absence of such lower acids may be determined by titration or gravimetrically. L. A. O'NEILL

**1375. Trace elements in edible oils. V. Separation and determination of iron using ion-exchange resins.** A. Vioque and M. del Pilar Villagrán (Inst. de la Grasa, Seville). *Grasas y Aceites*, 1957, **8** (4), 152-154.—The oil, dissolved in acetone, is passed through a cation-exchange column. The



acetone retained on the column is displaced by  $H_2O$ , and the Fe is eluted with 4 N HCl and estimated colorimetrically. Results obtained are similar to those from an ashing procedure, indicating that most if not all of the Fe in the oil is in an ionic form.

L. A. O'NEILL

**1376. The separation of the higher fatty acids and their glycerides by paper chromatography.** A. Holasek and J. Fries (Univ. Graz, Austria). *Mikrochim. Acta*, 1957, (3-4), 469-473 (in German).—For the detection of unsaturated fatty acids, they are oxidised with ozone and the resulting aldehydes are developed with pararosaniline-sulphurous acid. Fatty acids with one double bond give a blue spot, those with more double bonds give a red-violet spot. This principle has been successfully applied to the chromatography of the glycerides and other esters of the unsaturated fatty acids. A method is described for the separation of the mono-, di- and tri-glycerides from each other and from the free acid. The chromatogram is made on paper impregnated with liquid paraffin. Either 90% acetic acid or isopropyl alcohol-methanol- $H_2O$  mixtures may be used as mobile phases.

L. S. ADLER

**1377. Chromatographic analysis of mixtures of mono-, di- and tri-stearin containing mineral oil.** J. L. Revin, R. J. Meyer and T. Higuchi (Sch. of Pharm., Univ. of Wisconsin, Madison). *J. Amer. Oil Chem. Soc.*, 1957, **34** (6), 261-263.—Mono-, di- and tri-stearins can be separated individually, in the presence or absence of mineral oil, by elution chromatography on silica gel. The initial solvent is of low polarity, followed by a solvent or solvent mixtures of increasing polarity. Thus the component least strongly adsorbed, namely the triglyceride, becomes eluted first; and the component most strongly adsorbed, namely the monoglyceride, becomes eluted last. *Procedure*—Wet a column of silica gel (25 g) with isooctane. Dissolve the glyceride sample (1 to 2 g) in 100 ml of a 70% soln. of benzene in isooctane. Pipette a 10-ml aliquot on to the column, elute with 100% diisopropyl ether, followed by a soln. of 70% diethyl ether in isooctane, then by a soln. of 20% ethanol in diisopropyl ether. Any mineral oil is eluted before the tristearin.

G. HARDING

**1378. Colorimetric determination of ascorbic acid by the use of quadrivalent titanium salts.** Z. Gregorczyk (Zakl. Chem. Nieorg. Wydz. Farm., A.M. Wrocław). *Acta Polon. Pharm.*, 1957, **14** (3), 191-196.—The method of Hines and Boltz (*Anal. Chem.*, 1952, **24**, 947) for the determination of titanium has been adapted to the determination of ascorbic acid.

B. LAKE

**1379. Amperometric determination of ascorbic acid with potassium ferricyanide.** K. Hubicka (Katedry Chem. Nieorgan., Wydziału Mat.-Fiz.-Chem. UMCS, Lublin). *Ann. Univ. M. Curie-Skłodowska, A.A.*, 1955, **10** (4), 35-42.—The amperometric titration of ascorbic acid was successfully carried out by using 0.05 N  $K_4Fe(CN)_6$  and an applied voltage of 0.2 to 0.3 V, the acid soln. being buffered to pH 7 with  $KH_2PO_4$  or  $Na_2HPO_4$ . Rapid titrations in air (5 to 10 min.) gave results identical with those from titrations carried out in nitrogen. Additions of dextrose did not affect the results. The mean error was  $\pm 2\%$  and the method is considered to be particularly suitable for large numbers

of routine determinations. Titrations with  $K_2Cr_2O_7$  or  $K_2CrO_4$  gave more widely divergent results.

J. H. L. MESSING

See also Abstracts 1129, 1256, 1257, 1266, 1325.

### Sanitation

**1380. Rapid determination of phosphine in air.** J. P. Nelson and A. J. Milun (General Mills, Inc., Minneapolis, Minn., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1665-1666.—Air is forced through a column of silica gel impregnated with  $AgNO_3$ , at about 1.3 litres per min., until a black band at least 0.8 cm long is formed. The total quantity of gas passed is measured by a wet gas meter. The length of the coloured band is measured and the concn. determined from a calibration curve. The method is suitable for determining phosphine in air in the parts per  $10^6$  range, and gives a reproducibility of  $\pm 5\%$ . Interference is caused by  $AsH_3$ ,  $SbH_3$  and  $H_2S$ .

G. S. ROBERTS

**1381. Determination of trace amounts of lindane in air by infra-red spectrophotometry.** P. E. Braid and J. LeBoeuf (Dept. of Nat. Health and Welfare, Ottawa, Canada). *Anal. Chem.*, 1957, **29** (11), 1625-1627.—Lindane ( $\gamma$ -hexachlorocyclohexane,  $\leq 99\%$  purity) is characterised by three absorption peaks at 845, 698 and 685  $cm^{-1}$ , the last wavelength being used for quant. determination. The lindane is scrubbed from the air by means of an alumina column and is removed from this with hot isooctane, which is evaporated to a small volume before i.r. measurement. With air samples of 50 cu. ft., concn. of lindane from 1 to 100  $\mu g$  per cu. ft. can be measured. The estimated accuracy is  $\pm 3\%$  or  $\pm 0.3 \mu g$  per litre, whichever is greater.

G. P. COOK

**1382. Flame photometry in the analysis of natural waters. III. Determination of calcium.** P. Valori, M. Talenti and F. Savoini (Inst. of Hygiene, Univ., Rome). *Ric. Sci.*, 1957, **27** (6), 1901-1914.—The interference of various ions ( $Na^+$ ,  $K^+$ ,  $Li^+$ ,  $Mg^{2+}$ ,  $Cl^-$ ,  $SO_4^{2-}$ ) in the flame-photometric determination of Ca is avoided by the addition of a soln. containing a high concn. of the interfering ions and measurement of the difference in emission intensities at the Ca line (4227 Å) and at a nearby wavelength (4200 Å) not influenced by Ca; 1 p.p.m. of Ca may thereby be determined. A Beckman model DU spectrophotometer is used, with an oxy-hydrogen flame.

L. A. O'NEILL

**1383. Spectrochemical analysis of water.** R. Ko (G. E. Co., Hanford Atomic Prod. Oper., Richland, Wash.). *U.S. Atomic Energy Comm.*, Rep. HW-48770, 1957, 11 pp.—An evaporation-spectrographic method has been applied to the determination of traces of Fe, Ni, Cr, Zr, Ca, Mg, Cu, Al, Zn, Pb, Co and Mn in high-purity water. The sample is concentrated by evaporation with In as the internal standard. The residue is analysed by a graphite spark technique, with flat-top electrodes, with a precision of  $\pm 10\%$  (standard deviation of a single measurement).

NUCL. SCI. ABSTR.

**1384. Conductimetric method of determining hardness of water by means of EDTA (disodium salt).** G. B. Pasovskaya (Turkmen Medicinal Inst.,



Ashkhabad). *Zhur. Anal. Khim.*, 1957, **12** (4), 523-525.—After treatment with an ammoniacal buffer soln. (a mixture of 1 vol. of  $N NH_4Cl$  and 5 vol. of aq.  $NH_3$ ) the water is titrated conductometrically with 0.1  $N$  EDTA (disodium salt).

G. S. SMITH

**1385. Determination of uranium in natural waters.** L. L. Thatcher and F. B. Barker (U.S. Geological Survey, Washington, D.C., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1575-1578.—The fluorimetric method can be used for the routine determination of U in the solid residue from 10 ml of water. A high-carbonate flux (3 g) is used in a dilution procedure with spiking, and because of the high reflectivity of this large disc and the low concn. of U a correction is required for non-quenched light.

K. A. PROCTOR

**1386. Modified determination of radium in water.** F. B. Barker and L. L. Thatcher (U.S. Geological Survey, Washington, D.C., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1573-1575.—The method described can be used to detect as little as 0.1  $\mu\mu C$  of Ra by using 1-hr. counting times. It involves a barium sulphate carrier pptn., filtration through molecular filter membranes and counting of activities after an ageing period. The standard is  $^{226}Ra$  and the results indicate about 100 to 110% of the activity of the  $\alpha$ -emitting isotopes as  $^{223}Ra$ ,  $^{224}Ra$  and  $^{226}Ra$ .

K. A. PROCTOR

**1387. Use of ionites for the analysis of water and steam in the thermal power industry.** Yu. M. Kostrikin (F. E. Dzerzhinskii All-Union Thermo-technical Inst.). *Zavod. Lab.*, 1957, **23** (9), 1060-1063.—The effectiveness of certain ionites for the separation and determination of small amounts of Na, Ca, S, P, etc., in water is studied by means of radio-isotopes, and optimum conditions for the analysis are described.

G. S. SMITH

**1388. The occurrence of cadmium in sea water and in marine organisms and sediments.** J. B. Mullin and J. P. Riley. *J. Mar. Res.*, 1956, **15**, 103-122.—The procedure involves extraction with a  $CHCl_3$  soln. of dithizone in the presence of  $NH_4Cl$ , Na citrate and KCN to minimise the extraction of interfering elements. The Cd is then extracted from the  $CHCl_3$  layer with tartaric acid. The aq. layer is then made strongly alkaline with NaOH and the Cd is re-extracted with dilute dithizone in the presence of hydroxylamine and a small amount of cyanide. The optical density of the extract is measured at 518  $m\mu$ , the wavelength of max. absorption of Cd dithizonate. This method is capable of detecting less than 10  $\mu\mu g$  of Cd, and only Tl interferes.

WAT. POLLUT. ABSTR.

**1389. Determination of radio-strontium in effluent.** B. A. Loveridge and A. M. Thomas (A.E.R.E., Harwell, England). A.E.R.E. Report C/R 2294, 1957, 11 pp.—The procedure involves the addition of inactive strontium carrier to the sample ( $\approx 500$  ml), and the pptn. of the Sr, once as  $Sr_3(PO_4)_2$  and three times as  $Sr(NO_3)_2$ . After a  $Fe(OH)_3$  scavenge and two  $BaCrO_4$  scavenges, the Sr is pptd. as Sr oxalate, which is weighed and counted in a methane flow counter. All the counts are considered as being due to  $^{90}Sr$  and any  $^{90}Y$  that grows in before the counting is complete. The counter is calibrated by counting a source prepared by analys-

ing a standard  $^{90}Sr$ - $^{90}Y$  soln. in parallel with the sample. The possible occurrence of other radioactive nuclides of Sr and their daughter products in an effluent is considered. The analysis takes 2 hr. and the counting on less active samples takes  $\approx 1$  hr.

G. J. HUNTER

See also Abstract 1201.

## ■ Agriculture and Plant Biochemistry

**1390. Rapid determination of water in wet soils.** C. K. Hancock and R. L. Burdick (Texas Engng Exp. Sta., U.S.A.). *Soil Sci.*, 1957, **83**, 197-205.—The soil sample (10 g) is shaken with 100 ml (corr. to 30-0%) of a mixture of ethanol and acetone (70:30) (cf. Hancock and Hudgins, *Anal. Chem.*, 1954, **26**, 1738) and 10 g of NaCl (between 20- and 100-mesh). The sp. conductance of the extract is determined by a modified bridge meter by using 1000-cycle a.c. from a vacuum tube oscillator, a cathode ray tube as null indicator, and a temperature-compensating device. The standard deviation of determinations in three soils was  $\pm 0.06$  to  $0.22\%$ .

A. G. POLLARD

**1391. Use of an anion-exchange resin to eliminate anion interference in calcium determinations by flame photometry.** F. Adams and R. D. Rouse (Alabama Polytech. Inst., U.S.A.). *Soil Sci.*, 1957, **83**, 305-312.—In the presence of sulphate or phosphate, flame-photometric determinations of calcium give low results. Exchange of these anions for chloride by passing soil and plant extracts through a column of Dowex 2-X resin (Cl form) eliminates the interference.

A. G. POLLARD

**1392. Determination of the cation-exchange capacity of soils.** A. Wahhab and M. Ahmad (Agric. Coll. Lyallpur, W. Pakistan). *Soil Sci.*, 1957, **83**, 429-433.—The standard ammonium acetate method is modified by heating the mixture of soil (25 g) and neutral  $N$  ammonium acetate (200 ml), after shaking, at 70° for 2 hr. The soln. is cooled and filtered, and  $NH_3$  in the filtrate is determined by distillation with  $N$  NaOH (50 ml).

A. G. POLLARD

**1393. Amino-polysaccharides in soils: colorimetric determination of hexosamines in soil hydrolysates.** F. J. Stevenson (Illinois Univ., Urbana, U.S.A.). *Soil Sci.*, 1957, **83**, 113-122.—The soil (2 g) together with 10 ml of conc. HCl is placed in an all-glass reaction vessel of the Hamilton-Van Slyke type (*J. Biol. Chem.*, 1943, **150**, 231) and the mixture is shaken occasionally during 48 to 56 hr. Water (10 ml) is added and the reaction vessel is heated, with the side-arm open, at 100° for a few minutes. The side-arm is then closed and heating is continued until max. yield of hexosamines is obtained (6 to 18 hr., predetermined for each soil type). The reaction mixture is separated and the residue is washed three times centrifugally. The combined supernatant liquid and washings are evaporated to dryness at 40° and further dried over KOH. The residue is suspended in a minimum of water and washed on to a column (2.5 cm  $\times$  12.0 cm) of Amberlite IRA-400 (carbonate form). Humic acid, Fe and Al are retained on the resin and hexosamines are eluted with 0.02  $M$   $NaHCO_3$  and determined by Ehrlich's

method. Certain types of soil yield amino acid-sugar mixtures at this stage which interfere with colour formation. The hexamine soln. is then passed through Dowex-50 resin (H form) and subsequently eluted with 2 N HCl.

A. G. POLLARD

**1394. Micro-determination of organic compounds, using a new type of relaxational coulometer. III. Determination of plant-growth regulating substances.** K. Sykut (Katedry Chem. Nieorg. Wydziału Mat.-Fiz.-Chem. UMCS, Lublin). *Ann. Univ. M. Curie-Skłodowska, A.A.*, 1955, **10** (6), 53-60.—The method described in Part II (*cf. Anal. Abstr.*, 1958, **5**, 1265) was used for the determination of indol-3-yllactic acid (**I**) and other indole derivatives. It is concluded that for **I** the method is as good as colorimetric methods based on Salkowski's reaction, and is even better than these for the determination of other indole derivatives. J. H. L. MESSING

**1395. Hydrolytic method for the estimation of pyrethrin II in pyrethrum flowers.** L. W. Levy and H. Molia (Escuela Politéc. Nac., Quito, Ecuador). *Pyrethrum Post*, 1957, **4** (2), 22-24.—Dried ground flowers (5 g) are extracted for 8 to 10 hr. with light petroleum (boiling range 20° to 40°) in a Soxhlet apparatus and, after removal of the solvent, the residue is steam-distilled with 18 N H<sub>2</sub>SO<sub>4</sub> (20 ml), collecting 30 ml of distillate (which contains methanol formed by hydrolysis of pyrethrin II) in 10 ml of water in a 50-ml flask. This soln. is oxidised with KMnO<sub>4</sub>-H<sub>3</sub>PO<sub>4</sub> (destroying any excess with NaHSO<sub>3</sub>), and the resulting formaldehyde is determined photometrically after treatment of the soln. with chromotropic acid reagent. The extinction is measured at 560 mμ against a reagent blank. The method is rapid and reproducible, and is considered to be more specific than other available procedures. S. C. JOLLY

**1396. Polarographic behaviour of 1:1:1:2-tetrachloro-2:2-di-(p-chlorophenyl)ethane.** I. Rosenthal, G. J. Frisone and R. J. Lacoste (Rohm & Haas Co., Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1639-1641.—1:1:1:2-Tetrachloro-2:2-di-(p-chlorophenyl)ethane (**I**) gives two well-defined waves at approximately -0.2 and -1.85 V and a possible third wave at -2.6 V vs. the S.C.E. The first wave, which has been neglected by previous investigators, is a pH-independent, diffusion-controlled reduction in which *i*<sub>d</sub>/concn. is a constant. With this wave **I** can be determined in the presence of related compounds with a precision of ±2.2% relative over a concn. range of 1 to 10%; the optimum concn. is 10<sup>-3</sup> mM. The nature of the wave is discussed. G. P. COOK

**1397. Detection of E 605 [parathion] in biological material.** F. Scheibl and R. Stöhr (Med.-Chem. Inst., Univ. Innsbruck). *Mikrochim. Acta*, 1957, (3-4), 306-309 (in German).—The detection of p-nitrophenol has hitherto been regarded as sufficient proof for the presence of parathion in biological material. This detection rests on colour change from colourless to yellow when the medium is made alkaline, or on azo-dye formation, or on the presence of an absorption maximum at 405 mμ. Both the colour change and the azo-dye formation

may be shown by other organic compounds extractable from biological material, while detection of the absorption maximum depends on the availability of suitable apparatus. An analytical scheme is now described, in which the detection of parathion is based on confirmation of the presence of p-nitrophenol, sulphur and phosphoric acid.

L. S. ADLER

See also Abstracts 1112, 1171, 1237.

## 5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

### General

**1398. Automatic recording burette.** S. Gordon and C. Campbell (Pyrotechnics Chem. Res. Lab., Picatinny Arsenal, Dover, N.J., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1706-1708.—The pressure exerted by the liquid in a burette during a titration is utilised to actuate metal bellows. The movement of the bellows is converted to electrical signals by means of a linear variable differential transformer, and the signals may then be measured or recorded by electronic instruments. G. S. ROBERTS

**1399. Automatic solvent-extraction apparatus and method for uranium.** C. A. Kienberger (Oak Ridge Gaseous Diffusion Plant, Union Carbide Nuclear Co., Tenn., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1721-1722.—The laboratory apparatus described is designed for the purification of uranyl nitrate soln. from various impure sources, by an extraction process of 15 operations. The uranium oxide obtained from the final product is of >99.7% purity. It is suggested that the principle of the apparatus could be applied to large batch operations. G. S. ROBERTS

**1400. A small vacuum evaporator.** F. Petuely and N. Meixner (Biochem. Lab., Zool. Inst., Univ. Graz, Austria). *Mikrochim. Acta*, 1957, (3-4), 613-615 (in German).—The apparatus described permits the continuous concentration, without loss, by vacuum distillation of large volumes of liquid containing only small quantities of the required material. The apparatus takes up little space and is adaptable to any desired volume change. In the apparatus illustrated, concentration of any quantity to about 5 ml is possible at the rate of about 250 ml per hour. L. S. ADLER

**1401. Continuous fractional microsublimation in a divided or continuous sublimation tube.** B. Flaschenträger, S. M. Abdel-Wahhab and G. Habib-Labib (Med. Fac. and Biol. Fac., Univ. Alexandria, Egypt). *Mikrochim. Acta*, 1957, (3-4), 390-395 (in German).—Use is made of a Pyrex-glass tube, divided into four parts, which are joined by high vacuum ground joints. The high-vacuum sublimation is carried out in a heating block, through which the sections of the tube are pushed in turn. When the fractionation is complete, the fractions may be weighed in their individual tube sections. Equally good results may be obtained by using a continuous tube and breaking this into sections after the fractionation has taken place. L. S. ADLER

**1402. New automatic instruments for chromatographic analysis.** K. V. Chmutov and V. T. Avgul' (Inst. of Phys. Chem., Acad. Sci., USSR). *Zavod. Lab.*, 1957, **23** (9), 1115-1120.—A scheme for automatic chromatographic analysis, together with details of apparatus for mixing samples, drop counting, selecting portions of liquid, etc., is described.  
G. S. SMITH

**1403. Simple device for uniform application of liquid streaks for paper chromatography.** W. Merz (Univ. Graz, Austria). *Mikrochim. Acta*, 1957, (3-4), 474-479 (in German).—The device described enables micro amounts of liquid to be applied to the paper, and the amounts of materials obtained after elution of the chromatogram are sufficient for an exact identification.  
L. S. ADLER

**1404. Hydrophobisation of chromatography paper by ethylchlorosilane.** E. Kovács (Biochem. Inst. der Med. Univ., Szeged, Hungary). *Naturwissenschaften*, 1957, **44** (6), 181.—Impregnation of the paper with an 8% soln. of the ethylchlorosilane in hexanol gives complete hydrophobisation. With soln. of concn. <1% no hydrophobisation is obtained. With soln. from 1 to 7% the degree of hydrophobisation increases in a linear manner. As evaporation of the solvent is rapid it is a most convenient method for obtaining any desirable degree of hydrophobisation.  
E. KAWERAU

**1405. Quantitative method for the automatic application of millilitre quantities on to paper chromatograms.** L. Levenbook (Jefferson Med. Coll., Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1719-1720.—An apparatus is described which will deliver up to 0.5 ml of soln. on to chromatograph paper as a spot 4 to 5 mm in diameter. The apparatus comprises a hypodermic syringe with a piston controlled by a micrometer screw. The syringe is operated by a 2 r.p.m. synchronous motor, and delivers 0.01-ml aliquots, which are dried immediately on reaching the paper by a jet of warm air. The apparatus is so arranged that the rate of addition of soln. to the paper is equal to the rate of evaporation of the solvent.  
G. S. ROBERTS

**1406. Thermo-chromatography in the gas phase.** H. Kögler (VEB Kombinat "Otto Grotewahl," Böhlen, Leipzig). *Chem. Tech., Berlin*, 1957, **9** (7), 400-403.—A new technique of gas chromatography is characterised by a column with steadily rising temp. The absorption column (75 to 100 cm long, 5 to 8 mm wide) is filled with  $Al_2O_3$  silica gel or a high-boiling liquid on an inactive carrier. During the passage of the gas the temp. in the column is steadily raised by an electrical heating arrangement. Analysis of a mixture containing 19 components is much quicker than by the isothermic procedure hitherto used. Methods for the calculation of the results are given as well as graphs from tests. The method can be used for qualitative and quantitative analysis of hydrocarbon mixtures.  
C. A. CURTIS

**1407. Determination of the viscosity of liquids in c.g.s. units.** British Standards Institution (2 Park St., London). B.S. 188:1957, 63 pp.—In this revised Standard, the most important change is in the basis of calibration. Water at 20.00° is now the only standard for calibrating capillary viscometers, and the dynamic and kinematic viscosities

of water at 20° are to be accepted as 1.0020 centipoises and 1.0038 centistokes, respectively. Procedures and apparatus are described for the measurement of the viscosity of liquids exhibiting Newtonian flow.  
O. M. WHITTON

## Optical

**1408. A simple adaptor for the determination of spectra at low temperatures.** M. C. R. Symons and M. G. Townsend (Univ. of Southampton, Hants, England). *Unicam Spectrovision*, 1957, (4), 5-6.—An inexpensive Perspex and silica cell and cell-housing arrangement which completely replaces the removable cell-housing of the Unicam SP.500 spectrophotometer is described. A temperature of 155° K is readily achieved with the gas from heated liquid oxygen, and approximate optical density measurements have been made.  
E. G. CUMMINS

**1409. Statistical study on the homogeneity of zinc-base spectrographic standards.** R. C. Frank, J. E. Dallemard and D. L. Fry (General Motors Corp., Detroit, Mich., U.S.A.). *Spectrochim. Acta*, 1957, **9** (4), 323-331.—Any significant non-homogeneity of the minor constituents of the alloy was detected by a three-variable analysis of variance. The Duncan test was employed to find usable regions of homogeneity. Best possible results are obtained by confining sparking to a region midway between the centre and the edges of a segment.  
E. G. CUMMINS

**1410. Use of the SP.500 spectrophotometer in the near-infra-red region.** J. D. S. Goulden (Nat. Inst. Dairy Res., Reading Univ., England). *Unicam Spectrovision*, 1957, (4), 3-4.—The modifications to the wavelength scale and the introduction of a condenser system focused on to a Mullard 61SV lead sulphide detector are described. Details of the radiation-beam modulation device, a 17-slot sector wheel, rotating at 2825 r.p.m., are given. Good resolution of the 1.4- $\mu$ , 1.8- $\mu$  and 2.6- $\mu$  atmospheric absorption bands has been attained.  
E. G. CUMMINS

**1411. Determination of cell thickness by infra-red absorption.** J. Morcillo and J. A. Vinós ("A. de G. Rocasolano" Inst. Phys. Chem., Madrid). *An. Real Soc. Esp. Fis. Quim., B*, 1957, **53** (6), 411-418.—A rapid and general method for determining the thickness of infra-red liquid absorption cells by measuring optical densities of standard dilute soln. of  $CCl_4$  in  $CS_2$  is described. At 772  $cm^{-1}$ , the apparent mol. extinction of the strong band of  $CCl_4$  is practically constant and independent of the spectral slit width. The relation  $L = 0.03D/C$ , where  $L$  is the thickness of the cell in mm,  $D$  = optical density, and  $C$  = concn. in moles per litre, is applicable with an accuracy of  $\pm 0.0025$  mm.  
C. A. FINCH

## Electrical

**1412. Recent advances in electrophoretic separation methods for biologically important substances.** C. J. O. R. Morris (London Hosp. Med. Coll., London). *Phys. in Med. Biol.*, 1956, **1**, 3-17.

**1413. New technique of quantitative ultra-micro electrophoresis.** R. J. Wieme and M. Rabaey (Lab. of the Med. and Ophthalm. Clinic, Univ. of Ghent, Belgium). *Naturwissenschaften*, 1957, **44** (5), 112-113 (in English).—The whole operation is carried out on an ordinary microscope slide (2.5 cm  $\times$  7.5 cm). Agar (1.25% concn.) in barbitone buffer (pH 8.4) (ionic strength 0.05) is poured on to a slide to a thickness of 1.5 to 2.0 mm. A narrow groove is made across the agar surface which is used for holding the analytical material. Volumes of 1 to 0.1  $\mu$ l or of protein content of 0.1  $\mu$ g can be analysed. A direct current of 8 to 15 mA (150 V) is applied for 30 min. The slide is then covered with paper, dried at 37°, and treated for staining purposes like a tissue section. The slide is rendered translucent after the staining by drying, rinsing in toluene and mounting in Clarite. For quant. evaluation the slide can be scanned by a transmitted beam of light. Diagrams relating to soluble lens protein (human and reptile) are shown. E. KAWERAU

**1414. New method for polarographic standardisation.** J. T. Porter, II (Knolls Atomic Power Lab., Gen. Electric Co., Schenectady, N.Y., U.S.A.).—*Anal. Chem.*, 1957, **29** (11), 1638-1639.—A standardisation procedure is proposed which is claimed to be convenient in application, but not suitable for the most accurate work. Corrections are applied to changes in capillary characteristics detected by change in the diffusion current of a reference reduction soln. G. S. ROBERTS

**1415. Alternating current and square-wave polarography.** R. E. Hamm (Hanford Atomic Prod. Oper., Richland, Wash.). *U.S. Atomic Energy Comm.*, Rep. HW-45513, 1956, 16 pp.—Greater sensitivity and selectivity are achieved by using a polarographic technique in which an alternating sine or square-wave potential is superimposed upon the applied d.c. potential of the polarographic scan. As little as 0.005 g of U per litre can be determined in the presence of 0.05 M Fe<sup>3+</sup>.

NUCL. SCI. ABSTR.

**1416. Square-wave polarography. V. Organic adsorption-desorption waves.** G. C. Barker and A. W. Gardner (A.E.R.E., Harwell, England). A.E.R.E. Report C/R 1606, 1957, 26 pp.—The cause and effects of the adsorption of organic matter from solutions on to electrode surfaces are discussed in detail. An equivalent electrical circuit for the electrode-solution interface is derived and experimentally confirmed by using solutions containing octan-2-ol. The study of differential double-layer capacities by both d.c. and a.c. methods, the latter incorporating the square-wave impedance analyser, is described. E. G. CUMMINS

**1417. Voltammetry at controlled current. Automatic recording at solid electrodes.** R. N. Adams and J. H. Voorhies (Univ. of Kansas, Lawrence, U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1690-1694.—Automatic recording of current-scan polarograms in voltammetry at controlled current gives half-wave potentials in excellent agreement with accepted data. The technique should be useful for application to high-resistance polarography. K. A. PROCTOR

**1418. Ultra-micro methods of chemical analysis. Amperometric titration.** M. N. Petrikova and I. P. Alimarin (V. I. Vernadskii Inst. of Geochem. and Anal. Chem., USSR, Moscow). *Zhur. Anal. Khim.*, 1957, **12** (4), 462-465.—Apparatus for amperometric titration with a vibrating platinum electrode on a microscope stage of volumes of  $\approx 10^{-3}$  to  $10^{-4}$  ml is described. G. S. SMITH

**1419. Transistor amplifier for end-point indication of Karl Fischer titrations.** F. Oehme (Dtsch. Akad. Wissenschaften, Berlin). *Chem. Tech., Berlin*, 1957, **9** (6), 340-341.—A circuit diagram is given of a d.c. amplifier with transistors (unit the size of a matchbox) which permits the use of robust metering instruments giving full deflection with 3  $\mu$ A. A diagram of the whole titration and measuring assembly is provided. The apparatus is suitable for Karl Fischer water determinations (end-point indication according to "dead stop" method by Foulk and Bawden) and other polarisation titrations. It can also be used for iodine val. determinations and, as no excess of halogen is required, for selective saturation of double bonds. Great accuracy, low current consumption and no maintenance requirements are claimed. C. A. CURTIS

**1420. Physical apparatus and methods of measurement in radiochemical investigations.** Yu. A. Surkov and L. P. Moskaleva (V. I. Vernadskii Inst. of Geochem. and Anal. Chem., USSR). *Zavod. Lab.*, 1957, **23** (9), 1072-1080.—Apparatus for studying  $\alpha$ -activity (a scintillation  $\alpha$ -counter, chromatographic apparatus used with it, and an ionisation camera with an amplitude analyser of impulses),  $\beta$ -activity and  $\gamma$ -activity (a combined  $\beta$ - and  $\gamma$ -counter with two detectors and a luminescent  $\gamma$ -spectrometer) is described. G. S. SMITH

**1421. Evaluation of radiochemical separation procedures.** D. N. Sunderman and W. W. Meinke (Univ. Michigan, Ann Arbor, U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1578-1589.—Procedures for Ba, Sr, Ca and Ag have been critically evaluated and conditions are given for the optimum radiochemical separation of the three alkaline earths and for their systematic separation in tracer soln. Two standard and two new methods for the separation of radioactive Ag were studied, and one of them, isotopic exchange, is discussed in detail. It is specific for Ag, and quant. separation can be accomplished in 15 min. The use of ferric and lanthanum hydroxides as radioactive scavengers is described. K. A. PROCTOR

**1422. Ionisation-chamber assay of radioactive gases.** B. M. Tolbert (Radiation Lab., Univ., Berkeley, Calif.). *U.S. Atomic Energy Comm.*, Rep. UCRL-3499, 1956, 47 pp.—A practical review is presented on ionisation-chamber assay of radioactive gases. The subjects covered include ion-chamber theory, samples and sample preparation, combustion of organic compounds to CO<sub>2</sub>, design and construction of ion chambers, measurement of ion-chamber currents and approx. calibration data, and detailed procedures for <sup>14</sup>C and tritium assay. NUCL. SCI. ABSTR.

1423. Simple liquid scintillation counter for chemical analysis with radioactive tracers. W. Seaman (American Cyanamid Co., Bound Brook, N.J., U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1570-1573.—A simple, inexpensive and compact liquid scintillation counter with one photomultiplier and no refrigeration is described and is recommended for use when the necessary levels of activity are not disadvantageous. When used to determine naphthalene in tar-oil fractions by the isotope dilution method the coeff. of variation was about  $\pm 5\%$  (6 determinations). K. A. PROCTOR

1424. A surface-ionisation mass spectrometer for production control. M. W. Echo and T. D. Morgan (Phillips Petroleum Co., Idaho Falls, U.S.A.). *Anal. Chem.*, 1957, **29** (11), 1593-1595.—The instrument can be used for the measurement of the concn. and isotopic composition of uranium by an isotope dilution technique. On a control standard containing about 90% of  $^{235}\text{U}$  the error was  $\pm 0.11\%$  ( $P = 0.95$ ) for a single determination. The error in the ratio of  $^{235}\text{U}$  to  $^{238}\text{U}$  on 23 samples was about  $\pm 0.2\%$  ( $P = 0.95$ ).

K. A. PROCTOR



## ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

|                                            |                  |                                       |                                |
|--------------------------------------------|------------------|---------------------------------------|--------------------------------|
| alternating current . . . . .              | a.c.             | milli-equivalent . . . . .            | milli-equiv.                   |
| ampere . . . . .                           | amp.             | milligram . . . . .                   | mg                             |
| Ångström unit . . . . .                    | Å                | millilitre . . . . .                  | ml                             |
| anhydrous . . . . .                        | anhyd.           | millimetre . . . . .                  | mm                             |
| approximate, -ly . . . . .                 | approx.          | millimicrogram . . . . .              | mμg                            |
| aqueous . . . . .                          | aq.              | millimolar . . . . .                  | mM                             |
| atmosphere, -ic . . . . .                  | atm.             | millivolt . . . . .                   | mV                             |
| boiling-point . . . . .                    | b.p.             | minute (time) . . . . .               | min.                           |
| British thermal unit . . . . .             | B.Th.U.          | molar (concentration) . . . . .       | M                              |
| calorie (large) . . . . .                  | kg-cal.          | molecul-e, -ar . . . . .              | mol.                           |
| calorie (small) . . . . .                  | g-cal.           | normal (concentration) . . . . .      | N                              |
| centimetre . . . . .                       | cm               | optical rotation . . . . .            | α <sub>D</sub>                 |
| coefficient . . . . .                      | coeff.           | ounce . . . . .                       | oz                             |
| concentrated . . . . .                     | conc.            | parts per million . . . . .           | p.p.m.                         |
| concentration . . . . .                    | concn.           | per cent. . . . .                     | %                              |
| constant . . . . .                         | const.           | per cent. (vol. in vol.) . . . . .    | % (v/v)                        |
| corrected . . . . .                        | (corr.)          | per cent. (wt. in vol.) . . . . .     | % (w/v)                        |
| crystalline . . . . .                      | } cryst.         | per cent. (wt. in wt.) . . . . .      | % (w/w)                        |
| crystallised . . . . .                     |                  | potential difference . . . . .        | p.d.                           |
| cubic . . . . .                            | cu.              | precipitate (as a noun) . . . . .     | ppt.                           |
| current density . . . . .                  | c.d.             | precipitated . . . . .                | pptd.                          |
| cycles per second . . . . .                | c/s              | precipitating . . . . .               | pptg.                          |
| density . . . . .                          | ρ                | precipitation . . . . .               | pptn.                          |
| density, relative . . . . .                | d or wt. per ml  | preparation . . . . .                 | prep.                          |
| dilute . . . . .                           | dil.             | qualitative, -ly . . . . .            | qual.                          |
| direct current . . . . .                   | d.c.             | quantitative, -ly . . . . .           | quant.                         |
| distilled . . . . .                        | dist.            | recrystallised . . . . .              | recryst.                       |
| ethylenediaminetetra-acetic acid . . . . . | EDTA             | refractive index . . . . .            | n <sub>D</sub> <sup>20</sup>   |
| electromotive force . . . . .              | e.m.f.           | relative band speed . . . . .         | R <sub>b</sub>                 |
| equivalent . . . . .                       | equiv.           | relative humidity . . . . .           | r.h.                           |
| gram . . . . .                             | g                | revolutions per minute . . . . .      | r.p.m.                         |
| gram-molecule . . . . .                    | mole             | saponification value . . . . .        | sap. val.                      |
| half-wave potential . . . . .              | E <sub>1/2</sub> | saturated calomel electrode . . . . . | S.C.E.                         |
| hour . . . . .                             | hr.              | second (time) . . . . .               | sec.                           |
| hydrogen ion exponent . . . . .            | pH               | soluble . . . . .                     | sol.                           |
| infra-red . . . . .                        | i.r.             | solution . . . . .                    | soln.                          |
| insoluble . . . . .                        | insol.           | specific gravity . . . . .            | sp. gr.                        |
| international unit . . . . .               | i.u.             | specific rotation . . . . .           | [α] <sub>D</sub> <sup>20</sup> |
| kilogram . . . . .                         | kg               | square centimetre . . . . .           | sq. cm                         |
| kilovolt . . . . .                         | kV               | standard temp. and pressure . . . . . | s.t.p.                         |
| kilowatt . . . . .                         | kW               | temperature . . . . .                 | temp.                          |
| liquid . . . . .                           | liq.             | ultra-violet . . . . .                | u.v.                           |
| maxim-um, -a . . . . .                     | max.             | vapour density . . . . .              | v.d.                           |
| melting-point . . . . .                    | m.p.             | vapour pressure . . . . .             | v.p.                           |
| microgram . . . . .                        | μg (not γ)       | volt . . . . .                        | V                              |
| microlitre . . . . .                       | μl               | volume . . . . .                      | vol.                           |
| micromole . . . . .                        | μmole            | watt . . . . .                        | W                              |
| micron . . . . .                           | μ                | wavelength . . . . .                  | λ                              |
| milliampere . . . . .                      | mA               | weight . . . . .                      | wt.                            |

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

|                              |   |                                |   |
|------------------------------|---|--------------------------------|---|
| greater than . . . . .       | > | less than . . . . .            | < |
| not greater than . . . . .   | ≥ | not less than . . . . .        | ≤ |
| is proportional to . . . . . | ∝ | of the order of, approximately | ≈ |

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe<sup>II</sup>, Mo<sup>V</sup>. Substances in the ionic state are represented by Na<sup>+</sup>, Fe<sup>2+</sup>, Fe<sup>3+</sup>, etc., for cations and by Cl<sup>-</sup>, SO<sub>4</sub><sup>2-</sup>, PO<sub>4</sub><sup>3-</sup>, etc., for anions.

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|                                            |                         |                                       |                |
|--------------------------------------------|-------------------------|---------------------------------------|----------------|
| alternating current . . . . .              | a.c.                    | milli-equivalent . . . . .            | milli-equiv.   |
| ampere . . . . .                           | amp.                    | milligram . . . . .                   | mg             |
| Ångström unit . . . . .                    | Å                       | millilitre . . . . .                  | ml             |
| anhydrous . . . . .                        | anhyd.                  | millimetre . . . . .                  | mm             |
| approximate, -ly . . . . .                 | approx.                 | millimicrogram . . . . .              | m $\mu$ g      |
| aqueous . . . . .                          | aq.                     | millimolar . . . . .                  | mM             |
| atmospher-e, -ic . . . . .                 | atm.                    | millivolt . . . . .                   | mV             |
| boiling-point . . . . .                    | b.p.                    | minute (time) . . . . .               | min.           |
| British thermal unit . . . . .             | B.Th.U.                 | molar (concentration) . . . . .       | M              |
| calorie (large) . . . . .                  | kg-cal.                 | molecul-e, -ar . . . . .              | mol.           |
| calorie (small) . . . . .                  | g-cal.                  | normal (concentration) . . . . .      | N              |
| centimetre . . . . .                       | cm                      | optical rotation . . . . .            | $\alpha_D^t$   |
| coefficient . . . . .                      | coeff.                  | ounce . . . . .                       | oz             |
| concentrated . . . . .                     | conc.                   | parts per million . . . . .           | p.p.m.         |
| concentration . . . . .                    | concn.                  | per cent. . . . .                     | %              |
| constant . . . . .                         | const.                  | per cent. (vol. in vol.) . . . . .    | % (v/v)        |
| corrected . . . . .                        | (corr.)                 | per cent. (wt. in vol.) . . . . .     | % (w/v)        |
| crystalline . . . . .                      | } cryst.                | per cent. (wt. in wt.) . . . . .      | % (w/w)        |
| crystallised . . . . .                     |                         | potential difference . . . . .        | p.d.           |
| cubic . . . . .                            | cu.                     | precipitate (as a noun) . . . . .     | ppt.           |
| current density . . . . .                  | c.d.                    | precipitated . . . . .                | pptd.          |
| cycles per second . . . . .                | c/s                     | precipitating . . . . .               | pptg.          |
| density . . . . .                          | $\rho$                  | precipitation . . . . .               | pptn.          |
| density, relative . . . . .                | d or wt. per ml         | preparation . . . . .                 | prep.          |
| dilute . . . . .                           | dil.                    | qualitative, -ly . . . . .            | qual.          |
| direct current . . . . .                   | d.c.                    | quantitative, -ly . . . . .           | quant.         |
| distilled . . . . .                        | dist.                   | recrystallised . . . . .              | recryst.       |
| ethylenediaminetetra-acetic acid . . . . . | EDTA                    | refractive index . . . . .            | $n_D^t$        |
| electromotive force . . . . .              | e.m.f.                  | relative band speed . . . . .         | $R_f$          |
| equivalent . . . . .                       | equiv.                  | relative humidity . . . . .           | r.h.           |
| gram . . . . .                             | g                       | revolutions per minute . . . . .      | r.p.m.         |
| gram-molecule . . . . .                    | mole                    | saponification value . . . . .        | sap. val.      |
| half-wave potential . . . . .              | $E_{\frac{1}{2}}$       | saturated calomel electrode . . . . . | S.C.E.         |
| hour . . . . .                             | hr.                     | second (time) . . . . .               | sec.           |
| hydrogen ion exponent . . . . .            | pH                      | soluble . . . . .                     | sol.           |
| infra-red . . . . .                        | i.r.                    | solution . . . . .                    | soln.          |
| insoluble . . . . .                        | insol.                  | specific gravity . . . . .            | sp. gr.        |
| international unit . . . . .               | i.u.                    | specific rotation . . . . .           | $[\alpha]_D^t$ |
| kilogram . . . . .                         | kg                      | square centimetre . . . . .           | sq. cm         |
| kilovolt . . . . .                         | kV                      | standard temp. and pressure . . . . . | s.t.p.         |
| kilowatt . . . . .                         | kW                      | temperature . . . . .                 | temp.          |
| liquid . . . . .                           | liq.                    | ultra-violet . . . . .                | u.v.           |
| maxim-um, -a . . . . .                     | max.                    | vapour density . . . . .              | v.d.           |
| melting-point . . . . .                    | m.p.                    | vapour pressure . . . . .             | v.p.           |
| microgram . . . . .                        | $\mu$ g (not $\gamma$ ) | volt . . . . .                        | V              |
| microlitre . . . . .                       | $\mu$ l                 | volume . . . . .                      | vol.           |
| micromole . . . . .                        | $\mu$ mole              | watt . . . . .                        | W              |
| micron . . . . .                           | $\mu$                   | wavelength . . . . .                  | $\lambda$      |
| milliampere . . . . .                      | mA                      | weight . . . . .                      | wt.            |

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

|                              |           |                                          |           |
|------------------------------|-----------|------------------------------------------|-----------|
| greater than . . . . .       | >         | less than . . . . .                      | <         |
| not greater than . . . . .   | $\geq$    | not less than . . . . .                  | $\leq$    |
| is proportional to . . . . . | $\propto$ | of the order of, approximately . . . . . | $\approx$ |

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, e.g., Fe<sup>II</sup>, Mo<sup>V</sup>. Substances in the ionic state are represented by Na<sup>+</sup>, Fe<sup>2+</sup>, Fe<sup>3+</sup>, etc., for cations and by Cl<sup>-</sup>, SO<sub>4</sub><sup>2-</sup>, PO<sub>4</sub><sup>3-</sup>, etc., for anions.

## ANALYTICAL ABSTRACTS

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